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Steel and iron — Review of available methods of analysis

Aciers et fontes — Vue d'ensemble des méthodes d'analyse disponibles

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

This fourth edition cancels and replaces the third edition (ISO/TR 9769:1991), which has been technically revised to update all the standard methods for the determination of chemical composition of steel and iron.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Steel and iron — Review of available methods of analysis

1 Scope

This document gives guidelines for the determination of the chemical composition of steel and iron by reference to published International Standards, including their range of application and principles of the methods.

Graphical representation of the precision data precision data for the methods is given in Annex A.

The list of International Standards is summarized in Annex B.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
routine method
method calibrated against reference materials, certified reference materials or against standard solutions commercially available that is widely used for control purposes (day-to-day analysis)

4 International Standards for determining the chemical composition of steel and iron, their range of application and principles of the methods

4.1 Mono-elemental methods

4.1.1 Aluminium, Al

Document: ISO 9658:1990, *Steel — Determination of aluminium content — Flame atomic absorption spectrometric method*.

Range of application:

— determination of acid-soluble and/or total aluminium contents between a mass fraction of 0,005 % and a mass fraction of 0,20 % in non-alloyed steel.

Principle of the method:

a) dissolution of a test portion in dilute hydrochloric and nitric acids;

b) fusion of the acid-insoluble material with a mixture of orthoboric acid and potassium carbonate;

c) spraying of the solution into a dinitrogen monoxide-acetylene flame;

d) spectrometric measurement of the atomic absorption of the 309,3 nm spectral line emitted by an aluminium hollow cathode lamp.

4.1.2 Antimony, Sb

Document: ISO 10698:1994, *Steel — Determination of antimony content — Electrothermal atomic absorption spectrometric method*.

Range of application:

— determination of the antimony content between a mass fraction of 0,000 5 % and a mass fraction of 0,010 % in steel.

Principle of the method:

a) dissolution of a test portion in hydrochloric and nitric acids and dilution of the solution to a known volume;

b) introduction of a known volume of the solution into an electrothermal atomizer of an atomic absorption spectrometer;

c) measurement of the atomic absorption of the 217,6 nm spectral resonance line energy emitted by an antimony lamp, using background correction;

d) calibration by the standard additions technique.

4.1.3 Arsenic, As

Document: ISO 17058:2004, *Steel and iron — Determination of arsenic content — Spectrophotometric method*.

Range of application:

— determination of the arsenic content between 0,000 5 % (mass fraction) and 0,10 % (mass fraction) in steel and iron.

Principle of the method:

a) dissolution of a test portion in a hydrochloric acid and nitric acid mixture;

b) prolonged heating with sulfuric acid until white fumes are given off;

c) reduction of arsenic in the presence of hydrazine sulfate and potassium bromide, then separation by distillation as the trivalent chloride, absorbed in the nitric acid solution, in which the As(III) is oxidized back to the pentavalent state As(V);

d) formation of the molybdenum blue complex between ammonium molybdate and arsenic, which is reduced by hydrazine sulfate;

e) spectrophotometric measurement at a wavelength of approximately 840 nm.

4.1.4 Boron, B

4.1.4.1 Document: ISO 10153:1997, *Steel — Determination of boron content — Curcumin spectrophotometric method*.

Range of application:

— determination of the boron content between a mass fraction of 0,000 1 % and a mass fraction of 0,000 5 % only in unalloyed steel, and between a mass fraction of 0,000 5 % and a mass fraction of 0,012 % in steel.

Principle of the method:

a) dissolution of a test portion in hydrochloric and nitric acids;

b) decomposition of boron compounds (nitrides etc.) with orthophosphoric and sulfuric acids at a temperature of 290 °C;

c) formation of a coloured complex between orthoboric acid and curcumin in a buffered acetic medium;

d) spectrophotometric measurement at a wavelength of about 543 nm.

4.1.4.2 Document: ISO 13900:1997, *Steel — Determination of boron content — Curcumin spectrophotometric method after distillation*.

Range of application:

— determination of the boron content between a mass fraction of 0,000 05 % and a mass fraction of 0,001 0 % in steel.

Principle of the method:

a) dissolution of a test portion in hydrochloric and nitric acids;

b) decomposition of boron compounds (nitrides etc.) with orthophosphoric and sulfuric acids at a temperature of 290 °C;

c) distillation of the solution after the addition of methanol and collection of methylborate in a receiver containing sodium hydroxide solution;

d) evaporation of the solution to dryness; formation of a coloured complex between orthoboric acid and curcumin in a methanol medium;

e) spectrophotometric measurement at a wavelength of about 550 nm.

4.1.5 Calcium, Ca

4.1.5.1 Document: ISO 10697-1:1992, *Steel — Determination of calcium content by flame atomic absorption spectrometry — Part 1: Determination of acid-soluble calcium content*.

Range of application:

— determination of acid-soluble calcium content between a mass fraction of 0,000 5 % and a mass fraction of 0,003 % in steel.

Principle of the method:

a) dissolution of a test portion in hydrochloric and nitric acids;

b) addition of potassium chloride/lanthanum nitrate solution as a spectrochemical buffer;

c) spraying of the solution into a dinitrogen monoxide-acetylene flame;

d) spectrometric measurement of the atomic absorption of the 422,7 nm spectral line emitted by a calcium hollow cathode lamp.

4.1.5.2 Document: ISO 10697-2:1994, *Steel — Determination of calcium content by flame atomic absorption spectrometry — Part 2: Determination of total calcium content*.

Range of application:

— determination of the total calcium content between a mass fraction of 0,000 5 % and a mass fraction of 0,005 % in steel.

Principle of the method:

a) dissolution of a test portion in hydrochloric and nitric acids;

b) fusion of the acid-insoluble residue with sodium carbonate;

c) addition of potassium chloride/lanthanum nitrate solution as a spectrochemical buffer;

d) spraying of the solution into a dinitrogen monoxide-acetylene flame;

e) spectrometric measurement of the atomic absorption of the 422,7 nm spectral line emitted by a calcium hollow cathode lamp.

4.1.6 Carbon, C

4.1.6.1 Document: ISO 9556:1989, *Steel and iron — Determination of total carbon content — Infrared absorption method after combustion in an induction furnace*.

Range of application:

— determination of the total carbon content between a mass fraction of 0,003 % and a mass fraction of 4,5 % in steel and iron.

Principle of the method:

a) combustion of a test portion with accelerator at a high temperature in a high-frequency induction furnace in a current of pure oxygen; transformation of carbon into carbon dioxide and/or carbon monoxide;

b) measurement by infrared absorption of the carbon dioxide and/or carbon monoxide carried by a current of oxygen.

4.1.6.2 Document: ISO 15349-2:1999, *Unalloyed steel — Determination of low carbon content — Part 2: Infrared absorption method after combustion in an induction furnace (with preheating)*.

Range of application:

— determination of the carbon contents between a mass fraction of 0,000 3 % and a mass fraction of 0,010 % in unalloyed steel.

Principle of the method:

a) preheating of a test portion at low temperature and combustion of a test portion with accelerator at a high temperature in an induction furnace in a current of pure oxygen; transformation of carbon into carbon dioxide and/or carbon monoxide;

b) measurement of infrared absorption of the carbon dioxide or carbon dioxide/carbon monoxide evolved from steel and carried by a current of pure oxygen;

c) calibration graph is established using sucrose or calcium carbonate.

4.1.6.3 Document: ISO/TS 10719:2016, *Cast irons — Determination of non-combined carbon content — Infrared absorption method after combustion in an induction furnace*.

Range of application:

— determination of non-combined carbon content between a mass fraction of 1,0 % and a mass fraction of 3,0 % in cast irons.

NOTE Elements ordinarily present do not interfere. However, some alloyed cast irons, when extensively heat treated, contain carbides that are not soluble when using this method and can give high values for non-combined carbon.

Principle of the method:

a) decomposition of a test portion with nitric acid in the presence of methanol and treatment with hydrofluoric acid; removal of the non-combined carbon by filtering through a glass-fibre filter;

b) combustion of the glass-fibre filter containing the non-combined carbon in a flow of oxygen at a high temperature, using a high-frequency induction furnace, in the presence of pure iron and an accelerator; transformation of carbon into carbon dioxide and/or carbon monoxide;

c) measurement by infrared absorption of the carbon dioxide and/or carbon monoxide, carried by the current of oxygen.

4.1.7 Chromium, Cr

4.1.7.1 Document: ISO 4937:1986, *Steel and iron — Determination of chromium content — Potentiometric or visual titration method.*

Range of application:

— determination of chromium contents between a mass fraction of 0,25 % and a mass fraction of 35 % in steel and iron.

NOTE If vanadium is present, the visual titration is applicable only to test portions containing less than 3 mg of vanadium.

Principle of the method:

a) dissolution of a test portion with appropriate acids;

b) oxidation of chromium in an acid medium to chromium(VI) by ammonium peroxydisulfate in the presence of silver sulfate; reduction of manganese(VII) by hydrochloric acid;

c) reduction of chromium(VI) by ammonium iron(II) sulfate standard solution;

d) in the case of potentiometric detection, determination of the equivalence point by measurement of the potential variation when the ammonium iron(II) sulfate standard solution is being added;

e) in the case of visual detection, titration of the excess ammonium iron(II) sulfate by potassium permanganate standard solution, which also acts as the indicator.

4.1.7.2 Document: ISO 10138:1991, *Steel and iron — Determination of chromium content — Flame atomic absorption spectrometric method*.

Range of application:

— determination of chromium contents between a mass fraction of 0,002 % and a mass fraction of 2,0 % in non-alloy and low-alloy steels and iron.

Principle of the method:

a) dissolution of a test portion in hydrochloric acid, followed by oxidation with nitric acid;

b) filtration and ignition of the acid insoluble residue; removal of silica with hydrofluoric acid;

c) fusion of the residue with potassium hydrogen sulfate, extraction of the melt in acid and addition of the extract to the reserved filtrate;

d) spraying the solution into a dinitrogen monoxide-acetylene flame;

e) spectrometric measurement of the atomic absorption of the 357,9 nm or 425,4 nm spectral line emitted by a chromium hollow cathode lamp.

4.1.7.3 Document: ISO 15355:1999, *Steel and iron — Determination of chromium content — Indirect titration method*.

Range of application:

— determination of chromium content between a mass fraction of 1 % and a mass fraction of 35 % in steel and iron.

Vanadium contents in steel and iron should be less than a mass fraction of 1 % for chromium contents higher than a mass fraction of 10 % and less than a mass fraction of 0,2 % for chromium contents less than a mass fraction of 10 %.

Principle of the method:

a) fusion of the sample with sodium peroxide followed by acidification with sulfuric acid;

b) oxidization of chromium(III) to dichromate with peroxodisulfate using silver as a catalyst;

c) reduction of the dichromate with an excess of a solid iron(II) salt and potentiometric back titration of the excess with a dichromate solution;

d) any interference from vanadium is corrected mathematically.

4.1.8 Cobalt, Co

4.1.8.1 Document: ISO 11652:1997, *Steel and iron — Determination of cobalt content — Flame atomic absorption spectrometric method*.

Range of application:

— determination of the cobalt content between a mass fraction of 0,003 % and a mass fraction of 5,0 % in steel and iron.

Principle of the method:

a) dissolution of a test portion in hydrochloric, nitric and perchloric acids;

b) spraying of the solution into an air-acetylene flame;

c) spectrometric measurement of the atomic absorption of the 240,7 nm spectral line emitted by a cobalt hollow cathode lamp.

4.1.8.2 Document: ISO 11653:1997, *Steel — Determination of high cobalt content — Potentiometric titration method after separation by ion exchange*.

Range of application:

— determination of cobalt content between a mass fraction of 5,0 % and a mass fraction of 17,0 % in steel.

Principle of the method:

a) dissolution of a test portion in a mixture of nitric and hydrochloric acids;

b) separation of cobalt from interfering elements by selective elusion from an anion exchange column using hydrochloric acid;

c) oxidation with perchloric acid; removal of dissolved chlorine and oxygen with a stream of nitrogen;

d) addition of the test solution to a solution containing ammonium citrate, ammonium hydroxide and an excess of potassium hexacyanoferrate(lll) standard solution and titration of the excess with a standard cobalt solution using potentiometric end-point detection.

4.1.8.3 Document: ISO 13898-4:1997, *Steel and iron — Determination of nickel, copper and cobalt contents — Inductively coupled plasma atomic emission spectrometric method — Part 4: Determination of cobalt content*.

Range of application:

— determination of cobalt content between a mass fraction of 0,001 % and a mass fraction of 0,10 % in unalloyed steel and unalloyed iron.

Principle of the method:

a) dissolution of a test portion in a mixture of hydrochloric and nitric acids and dilution of the solution to known volume;

b) if necessary, addition of scandium or yttrium as internal standard;

c) nebulization of the solution into an inductively coupled plasma atomic emission spectrometer (ICP-AES) and measurement of the intensity of the emitted light from each element, or with emitted light from scandium or yttrium, simultaneously; examples of the analytical lines are given in ISO 13898-1:1997, Table 2.

4.1.9 Copper, Cu

4.1.9.1 Document: ISO 4943:1985, *Steel and cast iron — Determination of copper content — Flame atomic absorption spectrometric method*.

Range of application:

— determination of copper contents in the range of a mass fraction of 0,004 % to a mass fraction of 0,5 % in steel and cast iron.

Principle of the method:

a) dissolution of a test portion in a mixture of hydrochloric, nitric and perchloric acids;

b) spraying of the solution into an air-acetylene flame; spectrometric measurement of the atomic absorption of the 324,7 nm spectral line emitted by a copper hollow cathode lamp.

4.1.9.2 Document: ISO 4946:2016, *Steel and cast iron — Determination of copper — 2,2'-Biquinoline spectrophotometric method*.

Range of application:

— determination of copper mass fraction in the range of 0,02 % and 5 % in steel and cast iron.

Principle of the method:

a) dissolution of a test portion in appropriate acids;

b) fuming with perchloric acid to remove hydrochloric and nitric acids and dehydrate silicic acid;

c) reduction of copper(II) to copper(I) in hydrochloric acid solution by means of ascorbic acid; formation of a coloured compound of copper(I) with 2,2′-biquinoline;

d) spectrophotometric measurement at a wavelength of about 545 nm.

4.1.9.3 Document: ISO 13898-3:1997, *Steel and iron — Determination of nickel, copper and cobalt contents — Inductively coupled plasma atomic emission spectrometric method — Part 3: Determination of copper content*.

Range of application:

— determination of copper content between a mass fraction of 0,001 % and a mass fraction of 0,40 % in unalloyed steel and unalloyed iron.

Principle of the method:

a) dissolution of a test portion in a mixture of hydrochloric and nitric acids and dilution of the solution to known volume;

b) if necessary, addition of scandium or yttrium as internal standard;

c) nebulization of the solution into an ICP-AES and measurement of the intensity of the emitted light from each element, or with emitted light from scandium or yttrium, simultaneously; examples of the analytical lines are given in ISO 13898-1:1997, Table 2.

4.1.10 Manganese, Mn

4.1.10.1 Document: ISO 629:1982, *Steel and cast iron — Determination of manganese content — Spectrophotometric method*.

Range of application:

— determination of manganese contents between a mass fraction of 0,001 % and a mass fraction of 4 % in steel and cast iron.

Principle of the method:

a) dissolution of a test portion in sulfuric-phosphoric acid mixture and nitric acid; treatment of the test solution with perchloric acid; formation of the permanganate ion by sodium periodate;

b) spectrophotometric measurement of the test solution at wavelength of about 545 nm.

4.1.10.2 Document: ISO 10700:1994, *Steel and iron — Determination of manganese content — Flame atomic absorption spectrometric method*.

Range of application:

— determination of manganese content between a mass fraction of 0,002 % and a mass fraction of 2,0 % in steel and iron.

Principle of the method:

a) dissolution of a test portion in hydrochloric and nitric acids followed by evaporation with perchloric acid until white fumes appear;

b) spraying of the solution into an air-acetylene flame;

c) spectrometric measurement of the atomic absorption of the 279,5 nm spectral line emitted by a manganese hollow cathode lamp.

4.1.10.3 Document: ISO 10278:1995, *Steel — Determination of manganese content — Inductively coupled plasma atomic emission spectrometric method*.

Range of application:

— determination of the manganese content between a mass fraction of 0,002 % and a mass fraction of 1,5 % in unalloyed steels.

Principle of the method:

a) dissolution of a test portion in a mixture of hydrochloric and nitric acids and dilution of the solution to a known volume;

b) if necessary, addition of scandium or yttrium as an internal standard;

c) nebulization of the solution into an ICP-AES and measurement of the intensity of the emitted light from manganese at 257,61 nm and, optionally, emitted light from scandium at 361,38 nm or yttrium at 371,03 nm, simultaneously.

4.1.10.4 Document: ISO 18632:2018, *Alloyed steels — Determination of manganese — Potentiometric or visual titration method*.

Range of application:

— determination of the mass fraction of manganese between 2 % and 25 % in alloyed steels.

NOTE Vanadium and cerium interfere with the determination. If the mass fraction of cerium in the sample is less than 0,01 %, or the mass fraction of vanadium in the sample is less than 0,005 %, the interference is negligible, otherwise theoretical corrections are necessary.

Principle of the method:

a) dissolution of a test portion in appropriate acids;

b) addition of phosphoric acid; oxidation of manganese to manganese(III) in phosphoric acid medium with ammonium nitrate;

c) visual titration of manganese(III) with ferroammoniumdisulfate standard solution with N-phenylanthranilic acid as the indicator;

d) alternatively, potentiometric titration with ferroammoniumdisulfate standard solution; if the sample contains vanadium and cerium, the mass fraction of manganese is corrected.

4.1.11 Molybdenum, Mo

4.1.11.1 Document: ISO 4941:1994, *Steel and iron — Determination of molybdenum content — Thiocyanate spectrophotometric method*.

Range of application:

— determination of the molybdenum content between a mass fraction of 0,005 % and a mass fraction of 0,125 % in steel and iron.

NOTE 1 Vanadium and tungsten interfere with the measurement if, because of their contents, the V/Mo ratio is greater than 16 or the W/Mo ratio is greater than 8 (see NOTE 2).

NOTE 2 Greater V/Mo or W/Mo ratios (up to 300) can occur, but in such cases it is necessary to carry out the measurement very quickly after the extraction.

Principle of the method:

a) dissolution of a test portion in an appropriate mixture of acids and decomposition of the carbides by oxidation;

b) quantitative formation of a coloured compound of molybdenum, in the presence of thiocyanate, iron(ll) and/or copper(ll) ions and extraction of this compound using butyl acetate;

c) spectrophotometric measurement of the coloured compound at a wavelength of about 470 nm.

4.1.11.2 Document: ISO/TS 13899-1:2004, *Steel — Determination of Mo, Nb and W contents in alloyed steel — Inductively coupled plasma atomic emission spectrometric method — Part 1: Determination of Mo content*.

Range of application:

— determination of molybdenum content between a mass fraction of 0,03 % and a mass fraction of 8,5 % in steel.

Principle of the method:

a) dissolution of a test portion in a mixture of hydrochloric, nitric and phosphoric acid and fuming with a mixture of phosphoric and perchloric acids;

b) addition of hydrofluoric acid and, if necessary, an internal reference element and dilution of the solution to known volume;

c) nebulization of the solution into an ICP-AES and measurement of the intensity of the emitted light from molybdenum, or with emitted light from the internal reference element, simultaneously.

NOTE 1 The method uses a calibration graph based on a very close matrix matching of the calibration solutions to the sample and close bracketing of the contents around the approximate concentration of molybdenum in the sample to be analysed. The concentrations of all elements in the sample have to be, therefore, approximately known. If the concentrations are not known, the sample has to be analysed by some semi-quantitative method.

NOTE 2 The advantage with this procedure is that all possible interferences from the matrix are automatically compensated, which results in high accuracy. This is most important for spectral interferences, which can be severe in very highly alloyed steels. However, all possible interferences have to be kept on a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria specified in the method for the selected analytical lines.

4.1.12 Nickel, Ni

4.1.12.1 Document: ISO 4940:1985, *Steel and cast iron — Determination of nickel content — Flame atomic absorption spectrometric method*.

Range of application:

— determination of nickel contents in the range of a mass fraction of 0,002 % to a mass fraction of 0,5 % in steel and cast iron.

Principle of the method:

a) dissolution of a test portion in a mixture of appropriate acids followed by evaporation to fuming with perchloric acid;

b) spraying of the solution into an air-acetylene flame; spectrometric measurement of the atomic absorption of the 352,5 nm spectral line emitted by a nickel hollow cathode lamp.

NOTE 1 With some instruments it is not possible to obtain sufficient sensitivity at the wavelength of 352,5 nm for low concentrations of nickel near the bottom end of the application range. In such cases, the alternative wavelength of 232,0 nm is used.

NOTE 2 At the wavelength of 352,5 nm, the signal-to-noise ratio is higher than at a wavelength of 232,0 nm. In general, use of the 352,5 nm line will give better reproducibility.

4.1.12.2 Document: ISO 4938:2016, *Steel and iron — Determination of nickel content — Gravimetric or titrimetric method*.

Range of application:

— determination of nickel contents from a mass fraction of 1 % to a mass fraction of 30 % in steel and iron.

Principle of the method:

a) dissolution of a test portion with appropriate acids;

b) precipitation of the nickel as nickel-dimethylglyoxime;

  — cobalt, if present, is oxidized by potassium hexacyanoferrate(lll);

  — copper, if present with cobalt, preferably is removed by controlled potential electrolysis;

c) acid dissolution of the precipitate and filtration of the solution, followed by a second precipitation of the nickel as nickel dimethylglyoxime;

d) in the case of the gravimetric determination, weighing the dried dimethylglyoxime precipitate;

e) in the case of the titrimetric determination, acid dissolution of the precipitate, addition of excess EDTA.Na_2 solution and back titration of the excess EDTA.Na_2 by zinc solution using xylenol orange as an indicator;

f) in both cases, determination of residual nickel in the filtrate(s) by atomic absorption spectrometry.

4.1.12.3 Document: ISO 4939:2016, *Steel — Determination of nickel — Dimethylglyoxime spectrophotometric method*.

Range of application:

— determination of nickel mass fractions in the range of 0,10 % and 2,0 % in steel.

NOTE Cobalt, copper and manganese can cause interferences.

Principle of the method:

a) dissolution of a test portion in hydrochloric, nitric and perchloric acids;

b) formation of a coloured complex of nickel(lll) with dimethylglyoxime in ammoniacal solution containing iodine and potassium iodide;

c) spectrophotometric measurement at a wavelength of about 535 nm.

4.1.12.4 Document: ISO 13898-2:1997, *Steel and iron — Determination of nickel, copper and cobalt contents — Inductively coupled plasma atomic emission spectrometric method — Part 2: Determination of nickel content*.

Range of application:

— determination of nickel content between a mass fraction of 0,001 % and a mass fraction of 0,30 % in unalloyed steel and unalloyed iron.

Principle of the method:

a) dissolution of a test portion in a mixture of hydrochloric and nitric acids and dilution of the solution to known volume;

b) if necessary, addition of scandium or yttrium as internal standard;

c) nebulization of the solution into an ICP-AES and measurement of the intensity of the emitted light from each element, or with emitted light from scandium or yttrium, simultaneously; examples of the analytical lines are given in ISO 13898-1:1997, Table 2.

4.1.13 Niobium, Nb

4.1.13.1 Document: ISO 9441:1988, *Steel — Determination of niobium content — PAR spectrophotometric method*.

Range of application:

— determination of niobium content between a mass fraction of 0,005 % and a mass fraction of 1,3 % in steel.

Principle of the method:

a) dissolution of a test portion in hydrochloric acid followed by oxidation with hydrogen peroxide;

b) precipitation of niobium and tantalum with phenylarsonic acid, using zirconium as a carrier;

c) formation of a complex of niobium with 4(2-pyridylazo)-resorcinol (PAR) in a sodium tartrate medium buffered by sodium acetate solution adjusted to pH 6,3;

d) spectrophotometric measurement of the coloured compound at a wavelength of about 500 nm.

4.1.13.2 Document: ISO 13899-2:2005, *Steel — Determination of Mo, Nb and W contents in alloyed steel — Inductively coupled plasma atomic emission spectrometric method — Part 2: Determination of Nb content*.

Range of application:

— determination of niobium content contents between a mass fraction of 0,005 and a mass fraction of 5 % in steel.

Principle of the method:

a) the sample is dissolved in a hydrochloric, nitric and hydrofluoric acid mixture and fumed with a phosphoric and perchloric acid mixture; hydrofluoric acid and an internal standard element (if used) are added and the solution is diluted to known volume; the solution is filtered and nebulized into an inductively coupled plasma (ICP) and the intensity of the emitted light from each element is measured simultaneously with the light emitted from the internal standard element;

b) the method uses a calibration based on close matrix matching of the calibration solutions to the sample and close bracketing of the niobium content around the approximate concentration of niobium in the sample to be analysed; this compensates for matrix interferences and provides high accuracy, even in highly alloyed steels where spectral interferences can be severe; nonetheless, all interferences have to be kept to a minimum and it is therefore essential that the spectrometer used meets the performance criteria specified in the method for the selected analytical lines;

c) in order to accurately matrix match, it is necessary to know the concentration of all elements in the sample (to the nearest per cent); to this end, it may be necessary to carry out a preliminary analysis of the sample by some semi-quantitative method.

4.1.14 Nitrogen, N

4.1.14.1 Document: ISO 4945:2018, *Steel — Determination of nitrogen — Spectrophotometric method*.

Range of application:

— determination of nitrogen mass fraction between 0,000 6 % and 0,050 % in low alloy steels and between 0,010 % and 0,050 % in high alloy steels.

NOTE The method does not apply to samples containing silicon nitrides or having silicon contents higher than 0,6 %.

Principle of the method:

a) dissolution of a test portion in dilute sulfuric acid;

b) fuming of the acid-insoluble residue in sulfuric acid with potassium sulfate and copper(II) sulfate;

c) distillation of the solution made alkaline with sodium hydroxide, and collection of ammonia in a receiver containing diluted sulfuric acid;

d) formation of a blue-coloured complex between the ammonium ions and phenol in the presence of sodium hypochlorite and disodium pentacyanonitrosylferrate(III) (sodium nitroprusside);

e) spectrophotometric measurement of the complex at a wavelength of about 640 nm.

4.1.14.2 Document: ISO 10702:1993, *Steel and iron — Determination of nitrogen content — Titrimetric method after distillation*.

Range of application:

— determination of nitrogen contents from a mass fraction of 0,002 % to a mass fraction of 0,5 % in steel and iron;

— the method does not apply to samples containing silicon nitride.

Principle of the method:

a) dissolution of a test portion in hydrochloric acid followed by oxidation with hydrogen peroxide;

b) fuming of the acid-insoluble residue in sulfuric acid with potassium sulfate and copper(II) sulfate;

c) distillation of the solution made alkaline with sodium hydroxide, and collection of ammonia in a receiver containing orthoboric acid solution;

d) titration of the ammonium ion with amidosulfuric acid.

4.1.14.3 Document: ISO 10720:1997, *Steel and iron — Determination of nitrogen content — Thermal conductimetric method after fusion in a current of inert gas*.

Range of application:

— determination of nitrogen contents between a mass fraction of 0,000 8 % and a mass fraction of 0,5 % in steel and iron.

Principle of the method:

a) fusion of a test portion in a single-use graphite crucible under helium gas at a high temperature (e.g. 2 200 °C);

b) extraction of the nitrogen in the form of molecular nitrogen in the stream of helium;

c) separation from the other gaseous extracts and measurement by thermal conductimetric method.

4.1.14.4 Document: ISO 15351:1999, *Steel and iron — Determination of nitrogen content — Thermal conductimetric method after fusion in a current of inert gas (Routine method)*.

Range of application:

— determination of nitrogen contents between a mass fraction of 0,002 % and a mass fraction of 0,6 % in steel and iron.

Principle of the method:

a) fusion of a test portion in a single-use graphite crucible under helium gas at a high temperature (e.g. 2 200 °C);

b) extraction of the nitrogen in the form of molecular nitrogen in the stream of helium;

c) separation from the other gaseous extracts and measurement by thermal conductimetric method;

d) calibration graph established using steel or iron certified reference materials (CRMs).

NOTE The accuracy of the method is largely dependent on the accuracy of the method used to certify the nitrogen concentration of CRMs as well as on their homogeneity. The calibration specimens used are ECRM, NIST, JK, JSS, CMSI and other national or international CRMs.

4.1.15 Oxygen, O

Document: ISO 17053:2005, *Steel and iron — Determination of oxygen — Infrared method after fusion under inert gas*.

Range of application:

— determination of mass fractions of oxygen between 0,000 75 % and 0,01 % in steel and iron.

Principle of the method:

a) fusion of a test portion in a single-use graphite crucible under helium gas at a minimum temperature of 2 000 °C; combination of the oxygen from the sample with carbon from the crucible to form carbon monoxide; eventually, transformation of carbon monoxide into carbon dioxide;

b) measurement of infrared absorption of the carbon monoxide or dioxide, against a calibration curve made of potassium nitrate.

4.1.16 Phosphorus, P

Document: ISO 10714:1992, *Steel and iron — Determination of phosphorus content — Phosphovanadomolybdate spectrophotometric method*.

Range of application:

— determination of phosphorus contents between a mass fraction of 0,001 0 % and a mass fraction of 1,0 % in steel and iron.

NOTE Arsenic, hafnium, niobium, tantalum, titanium, and tungsten interfere in determining phosphorus, but the interferences can be partially overcome by formation of complexes and use of small quantities of test portion. Depending on the concentration of the interfering elements, the application ranges and test portions given in ISO 10714:1992, Table 1, apply.

Principle of the method:

a) dissolution of a test portion in an oxidizing acid mixture;

b) fuming with perchloric acid and removal of chromium as volatile chromyl chloride;

c) complexing of silicon and the refractory elements with hydrofluoric acid and complexing of the excess of hydrofluoric acid with orthoboric acid;

d) conversion of phosphorus to phosphovanado-molybdate in perchloric and nitric acid solution;

e) extraction of phosphovanadomolybdate by 4-methyl-2-pentanone with citric acid present to complex arsenic;

f) spectrophotometric measurement at a wavelength of 355 nm.

4.1.17 Sulfur, S

4.1.17.1 Document: ISO 671:1982, *Steel and cast iron — Determination of sulphur content — Combustion titrimetric method*.

Range of application:

— determination of sulfur in steel and cast iron, after combustion of the test portion in a current of oxygen.

Principle of the method:

a) combustion of a test portion at a high temperature (1 450 °C) in a current of pure oxygen, if necessary in the presence of a metal flux;

b) transformation of sulfur into sulfur dioxide; absorption of the gases resulting from combustion, freed from oxide dust, by a solution of potassium sulfate and hydrogen peroxide;

c) titration of the absorbent solution with a standard sodium borate solution.

4.1.17.2 Document: ISO 4934:2003, *Steel and iron — Determination of sulfur content — Gravimetric method*.

Range of application:

— determination of sulfur content between a mass fraction of 0,003 % and a mass fraction of 0,35 % in steels and iron, excluding steels containing selenium.

The method is particularly suitable as a reference method for the standardization of samples on which certified standard values are to be established.

Principle of the method:

a) dissolution of a test portion in dilute nitric acid in the presence of bromine, or in the mixed acid of nitric acid and hydrochloric acid in the presence of bromine (with the aid of an appropriate device to prevent sulfur losses);

b) addition of perchloric acid and evaporation of the solution until white fumes of perchloric acid are evolved;

c) filtration of the solution and removal of the dehydrates of silicon, tungsten, niobium, etc;

d) addition of a determined quantity of sulfate ions to aid precipitation;

e) chromatographic separation of the sulfate ions from the test solution by adsorption on an alumina column, and elution using an ammonium hydroxide solution;

f) precipitation of the sulfate ions as barium sulfate under controlled conditions and filtering, washing, heating and weighing.

4.1.17.3 Document: ISO 4935:1989, *Steel and iron — Determination of sulfur content — Infrared absorption method after combustion in an induction furnace*.

Range of application:

— determination of sulfur content between a mass fraction of 0,002 % and a mass fraction of 0,10 % in steel and iron.

Principle of the method:

a) combustion of a test portion with accelerator at a high temperature in a high-frequency induction furnace in a current of pure oxygen; transformation of sulfur into sulfur dioxide;

b) measurement by infrared absorption of the sulfur dioxide carried by a current of oxygen.

4.1.17.4 Document: ISO 10701:1994, *Steel and iron — Determination of sulfur content — Methylene blue spectrophotometric method*.

Range of application:

— determination of sulfur contents between a mass fraction of 0,000 3 % and a mass fraction of 0,010 % in steel and iron.

NOTE Niobium, silicon, tantalum and titanium interfere in the determination of sulfur. Depending on the concentration of the interfering elements, the application ranges and test portion masses given in ISO 10701:1994, Table 1, apply.

Principle of the method:

a) dissolution of a test portion in a mixture of hydrochloric and nitric acids;

b) evaporation with perchloric acid until white fumes appear to remove hydrochloric and nitric acids;

c) dissolution of the salts in hydrochloric acid; evolution of hydrogen sulfide by reducing with a mixture of hydroiodic and hypophosphorous acids in a nitrogen atmosphere, distillation, and absorption into zinc acetate solution;

d) formation of methylene blue by reacting with N,N-dimethyl-p-phenylenediamine and iron(lll) solution;

e) spectrophotometric measurement at a wavelength of about 665 nm.

4.1.17.5 Document: ISO 13902:1997, *Steel and iron — Determination of high sulfur content — Infrared absorption method after combustion in an induction furnace*.

Range of application:

— determination of sulfur content between a mass fraction of 0,10 % and a mass fraction of 0,35 % in steel and iron.

Principle of the method:

a) combustion of a test portion with accelerator at a high temperature in a high-frequency induction furnace in a current of pure oxygen;

b) transformation of sulfur into sulfur dioxide;

c) measurement by infrared absorption of the sulfur dioxide carried by a current of oxygen.

4.1.18 Silicon, Si

4.1.18.1 Document: ISO 439:1994, *Steel and iron — Determination of total silicon content — Gravimetric method*.

Range of application:

— determination of the total silicon content between a mass fraction of 0,10 % and a mass fraction of 5,0 % in steel and iron.

NOTE For samples containing molybdenum, niobium, tantalum, titanium, tungsten, zirconium or high levels of chromium, the results are less precise than for unalloyed steels.

Principle of the method:

a) attack of a test portion by hydrochloric and nitric acids;

b) conversion of acid-soluble silicon compounds to hydrated silicon dioxide by evaporation with perchloric acid until white fumes appear; filtration of the hydrated silicon dioxide and acid-insoluble silicon compounds, ignition to form impure silicon dioxide and then weighing;

c) treatment of the ignited residue with hydrofluoric and sulfuric acids, followed by ignition and weighing.

4.1.18.2 Document: ISO 4829-1:2018, *Steel and cast iron — Determination of total silicon contents — Reduced molybdosilicate spectrophotometric method — Part 1: Silicon contents between 0,05 % and 1,0 %*.

Range of application:

— determination of total silicon mass fraction between 0,05 % and 1,0 % in steel and cast iron.

Principle of the method:

a) dissolution of a test portion in an acid mixture appropriate to the alloy composition;

b) fusion of the acid-insoluble residue with sodium peroxide; formation of the oxidized molybdosilicate (yellow) complex in weak acid solution;

c) selective reduction of the molybdosilicate complex to a blue complex with ascorbic acid, after increasing the sulfuric acid concentration and adding oxalic acid to prevent the interference of phosphorus, arsenic and vanadium;

d) spectrophotometric measurement of the reduced blue complex at a wavelength of about 810 nm.

4.1.18.3 Document: ISO 4829-2:2016, *Steels — Determination of total silicon contents — Reduced molybdosilicate spectrophotometric method — Part 2: Silicon contents between 0,01 % and 0,05 %*.

Range of application:

— determination of total silicon contents between a mass fraction of 0,01 % and a mass fraction of 0,05 % in steels.

Principle of the method:

a) dissolution of a test portion in a hydrochloric/nitric acids mixture;

b) fusion of the acid-insoluble residue with sodium peroxide; formation of the oxidized molybdosilicate (yellow) complex in weak acid solution;

c) selective reduction of the molybdosilicate complex to a blue complex with ascorbic acid, after increasing the sulfuric acid concentration and adding oxalic acid to prevent the interference of phosphorus, arsenic and vanadium;

d) spectrophotometric measurement of the reduced blue complex at a wavelength of about 810 nm.

4.1.18.4 Document: ISO/TR 17055:2002, *Steel — Determination of silicon content — Inductively coupled plasma atomic emission spectrometric method*.

Range of application:

— determination of silicon content of a mass fraction 0,02 % to a mass fraction of 5 % in steel by means of ICP emission spectrometry.

Principle of the method:

a) the sample is dissolved in a hydrochloric, nitric and hydrofluoric acid mixture;

b) an internal standard element is added and the solution is diluted to known volume;

c) the solution is nebulized into an ICP and the intensity of the emitted light from each element is measured simultaneously with the light emitted from the internal standard element.

4.1.19 Tin, Sn

Document: ISO 15353:2001, *Steel and iron — Determination of tin content — Flame atomic absorption spectrometric method (extraction as Sn-SCN)*.

Range of application:

— determination of tin contents in the range 0,001 % by mass and 0,1 % by mass in steel and iron.

Principle of the method:

a) dissolution of a test portion in hydrochloric and nitric acids; formation of Sn-SCN complex and extraction of the complex into 4-methyl-2-pentanone (isobutyl methyl ketone);

b) aspiration of the organic solution into a dinitrogen monoxide-acetylene flame; spectrometric measurement of the atomic absorption of the 224,6 nm spectral line emitted by a tin hollow cathode lamp;

c) high purity tin metal dissolved as standard solution is used as reference material for calibration graphs.

4.1.20 Titanium, Ti

Document: ISO 10280:1991, *Steel and iron — Determination of titanium content — Diantipyrylmethane spectrometric method*.

Range of application:

— determination of titanium contents between a mass fraction of 0,002 % and a mass fraction of 0,80 % in steel and iron.

Principle of the method:

a) dissolution of a test portion in hydrochloric, nitric and sulfuric acids;

b) fusion of the residue with potassium hydrogen sulfate;

c) formation of a yellow complex with 4,4′-diantipyrylmethane;

d) spectrophotometric measurement of the coloured complex at a wavelength of about 385 nm.

4.1.21 Tungsten, W

Document: ISO/TS 13899-3:2005, *Steel — Determination of Mo, Nb and W contents in alloyed steel — Inductively coupled plasma atomic emission spectrometric method — Part 3: Determination of W content.*

Range of application:

— determination of tungsten content between a mass fraction of 0,1 % and a mass fraction of 20 % in steel.

Principle of the method:

a) the sample is dissolved in a hydrochloric, nitric and hydrofluoric acid mixture and fumed with a phosphoric and perchloric acid mixture; hydrofluoric acid and an internal standard element (if used) are added and the solution is diluted to known volume; the solution is filtered and nebulized into an ICP spectrometer and the intensity of the emitted light from the element is measured simultaneously with the light emitted from the internal standard element;

b) the method uses a calibration based on close matrix matching of the calibration solutions to the sample and close bracketing of the tungsten content around the approximate concentration of tungsten in the sample to be analysed; this compensates for matrix interferences and provides high accuracy, even in highly alloyed steels where spectral interferences can be severe; nonetheless, all interferences have to be kept to a minimum and it is therefore essential that the spectrometer used meets the performance criteria specified in the method for the selected analytical lines;

c) in order to accurately matrix match, it is necessary to know the content of all elements in the sample (to the nearest %); to this end, it may be necessary to carry out a preliminary analysis of the sample by a semi-quantitative method.

4.1.22 Vanadium, V

4.1.22.1 Document: ISO 4942:2016, *Steels and irons — Determination of vanadium content — N-BPHA spectrophotometric method*.

Range of application:

— determination of vanadium contents between a mass fraction of 0,005 % and a mass fraction of 0,50 % in steels and cast irons.

Principle of the method:

a) dissolution of a test portion with appropriate acids;

b) addition of orthophosphoric acid to an aliquot of the sample solution to prevent the interference of iron, and addition of potassium permanganate to oxidize vanadium to the pentavalent state;

c) selective reduction of excess permanganate by sodium nitrite, in the presence of urea and treatment with N-BPHA and hydrochloric acid to form a complex, followed by extraction of the complex with trichloromethane;

d) spectrophotometric measurement of the absorbance at approximately 535 nm.

4.1.22.2 Document: ISO 4947:1986, *Steel and cast iron — Determination of vanadium content — Potentiometric titration method*.

Range of application:

— determination of vanadium contents between a mass fraction of 0,04 % and a mass fraction of 2 % in steel and cast iron.

Principle of the method:

a) dissolution of a test portion with appropriate acids; addition of hydrofluoric acid to keep tungsten in solution;

b) oxidation of chromium and vanadium by potassium peroxy-disulfate; partial oxidation of chromium;

c) while checking the potential of the solution:

  - reduction of chromium(VI) and vanadium(V) by ammonium iron(II) sulfate;
  - oxidation of vanadium by slight excess of potassium permanganate; reduction of the excess permanganate by sodium nitrite, and reduction of the excess sodium nitrite by sulfamic acid;

d) potentiometric titration of vanadium by ammonium iron(II) sulfate standard solution.

4.1.22.3 Document: ISO 9647:1989, *Steel and iron — Determination of vanadium content — Flame atomic absorption spectrometric method*.

Range of application:

- determination of vanadium contents between a mass fraction of 0,005 % and a mass fraction of 1,0 % in steel and iron, provided that the tungsten content in the test portion is not higher than 10 mg and/or the titanium content is not higher than 5 mg.

Principle of the method:

a) dissolution of a test portion in hydrochloric, nitric and perchloric acids;

b) addition of aluminium solution as spectrochemical buffer;

c) spraying the solution into a dinitrogen monoxide-acetylene flame;

d) spectrometric measurement of the atomic absorption of the 318,4 nm spectral line emitted by a vanadium hollow cathode lamp.

4.2 Multi-elemental methods

4.2.1 Calcium, Ca; Magnesium, Mg

Document: ISO 13933:2014, *Steel and iron — Determination of calcium and magnesium — Inductively coupled plasma atomic emission spectrometric method*.

Range of application:

- determination of calcium content (mass fraction) in the range of 0,000 5 % to 0,006 %;
- determination of magnesium contents (mass fraction) in the range of 0,000 5 % to 0,20 % in iron, cast iron, steel, and alloyed steel.

Principle of the method:

a) a test portion is dissolved in a hydrochloric, nitric and hydrofluoric acid mixture and fumed with perchloric acid;

b) hydrochloric acid, nitric acid, and an internal standard element (if used) are added and the solution is diluted to a known volume;

c) the solution is filtered, if necessary, nebulized into an ICP and the intensity of the emitted light from each element is measured simultaneously with the intensity of the light emitted by the internal standard element.

4.2.2 Carbon, C; Sulfur, S

Document: ISO 15350:2000, *Steel and iron — Determination of total carbon and sulfur content — Infrared absorption method after combustion in an induction furnace (routine method).*

Range of application:

- determination of carbon contents from a mass fraction of 0,005 % to a mass fraction of 4,3 %;
- determination of sulfur contents from a mass fraction of 0,000 5 % to a mass fraction of 0,33 %.

Principle of the method.

a) the carbon is converted to carbon monoxide and/or carbon dioxide by combustion in a stream of oxygen; measurement is by infrared absorption of the carbon monoxide and carbon dioxide carried by a current of oxygen;

b) the sulfur is converted to sulfur dioxide by combustion in a stream of oxygen; measurement is by infrared absorption of the sulfur dioxide carried by a current of oxygen.

4.2.3 Tin, Sn; Antimony, Sb; Cerium, Ce; Lead, Pb; Bismuth, Bi

Document: ISO 16918-1:2009, *Steel and iron — Determination of nine elements by the inductively coupled plasma mass spectrometric method — Part 1: Determination of tin, antimony, cerium, lead and bismuth.*

Range of application:

- determination of tin contents from: 5 µg/g to 200 µg/g;
- determination of antimony contents from: 1 µg/g to 200 µg/g;
- determination of cerium contents from: 10 µg/g to 1 000 µg/g;
- determination of lead contents from: 0,5 µg/g to 100 µg/g;
- determination of bismuth contents from: 0,3 µg/g to 30 µg/g.

Principle of the method:

a) a test portion is dissolved in an acid-mixture of hydrochloric acid, nitric acid and hydrofluoric acid using either a microwave-assisted system or a traditional hot plate;

b) diluted wet-digested samples are introduced into an inductively coupled plasma mass spectrometer (ICP-MS), via a peristaltic pump; simultaneous measurements of the intensities of elements with atomic mass units of concern (mass spectra) are carried out using ICP-MS techniques;

c) calibration blank and calibration solutions are matrix-matched with the major elements of steel, and mineral acids are used for wet-digestion;

d) internal standards are used throughout in order to compensate for any instrument drift.

4.2.4 Chromium, Cr; Cobalt, Co; Copper, Cu; Manganese, Mn; Molybdenum, Mo; Nickel, Ni; Niobium, Nb; Phosphorus, P; Silicon, Si; Titanium, Ti; Vanadium, V

Document: ISO 17054:2010, *Routine method for analysis of high alloy steel by X-ray fluorescence spectrometry (XRF) by using a near-by technique.*

Range of application:

- determination of chromium contents from a mass fraction of 10 % to a mass fraction of 25 %;
- determination of cobalt contents from a mass fraction of 0,015 % to a mass fraction of 0,30 %;

- determination of copper contents from a mass fraction of 0,02 % to a mass fraction of 1,5 %;
- determination of manganese contents from a mass fraction of 0,05 % to a mass fraction of 5,0 %;
- determination of molybdenum contents from a mass fraction of 0,1 % to a mass fraction of 6,5 %;
- determination of nickel contents from a mass fraction of 0,1 % to a mass fraction of 30 %;
- determination of niobium contents from a mass fraction of 0,05 % to a mass fraction of 1,0 %;
- determination of phosphorus contents from a mass fraction of 0,005 % to a mass fraction of 0,035 %;
- determination of silicon contents from a mass fraction of 0,05 % to a mass fraction of 1,5 %;
- determination of titanium contents from a mass fraction of 0,015 % to a mass fraction of 0,50 %;
- determination of vanadium contents from a mass fraction of 0,015 % to a mass fraction of 0,15 %.

The method is applicable to analysis of either chill-cast or wrought samples having a diameter of at least 25 mm and with a carbon concentration of less than 0,3 % (see NOTE). Other elements have a concentration below 0,2 %.

NOTE High carbon concentrations, in combination with high Mo and Cr concentrations, could have undesirable structural effects on the sample and could affect the determination of phosphorus and chromium, in particular.

Principle of the method:

a) the sample is finished to a clean uniform surface and then irradiated by an X-ray beam of high energy;

b) the secondary X-rays produced are dispersed by means of crystals and the intensities are measured by detectors at selected characteristic wavelengths; the measuring time is set to reach below a specified counting statistical error;

c) preliminary concentrations of the elements are determined by relating the measured intensities of unknown samples to analytical curves prepared from reference materials (CRMs or RMs) of known compositions; the final concentrations are calculated by using the results obtained by measuring a CRM of the same grade;

d) the correction is made for the elements of interest by using the difference between the certified value and the value obtained during the measurement of the CRM (the “near-by technique”);

e) a fixed channel or a sequential system may be used to provide simultaneous or sequential determinations of element concentrations.

4.2.5 Zinc, Zn; Aluminium, Al; Nickel, Ni; Iron, Fe; Silicon, Si; Lead, Pb

Document: ISO 17925:2004, *Zinc and/or aluminium based coatings on steel — Determination of coating mass per unit area and chemical composition — Gravimetry, inductively coupled plasma atomic emission* spectrometry *and flame atomic absorption spectrometry*.

Range of application:

- determination of zinc contents between a mass fraction of 40 % and a mass fraction of 100 %;
- determination of aluminium contents between a mass fraction of 0,02 % and a mass fraction of 60 %;
- determination of nickel contents between a mass fraction of 7 % and a mass fraction of 20 %;
- determination of iron contents between a mass fraction of 0,2 % and a mass fraction of 20 %;
- determination of silicon contents between a mass fraction of 0,2 % and a mass fraction of 10 %;
- determination of lead contents between a mass fraction of 0,005 % and a mass fraction of 2 %.

NOTE 1 This method is applicable for the determination of the coating mass per unit area by gravimetry and the chemical composition of one side-surface of zinc- and/or aluminium-based coatings on steel by means of ICP-AES or FAAS. For example, the test method applies for zinc and/or aluminium based coatings on steel such as galvanize (hot-dip and electrolytic), galvaneal (hot-dip), zinc-nickel electrolytic, zinc-5 % aluminium coating (hot-dip) and zinc-55 % aluminium coating (hot-dip). Galvanizing gives a pure zinc coating. Galvanealling gives a zinc-iron alloyed coating. Zinc-nickel electrolytic methods give zinc-nickel alloyed coatings.

NOTE 2 For example, the applicable elements for these products are as follows: galvanizing is specified for iron and aluminium; galvanealling is specified for zinc, iron and aluminium; zinc-nickel electrolytic methods are specified for zinc, iron and nickel; zinc-5 % aluminium coating is specified for zinc, iron, aluminium and silicon; zinc-55 % aluminium is specified for zinc, iron, aluminium and silicon.

Principle of the method:

a) stripping the coating on one side on the base steel in a mixture of hydrochloric acid solution containing an inhibitor to prevent the attack on the base steel; determination of the mass per unit area of coating from the mass difference of the specimen before and after stripping; calculating the coating mass as the mass difference divided by the specimen's surface area;

b) dilution of the stripped solution of the coating on one side; filtration and nebulization of the solution into an ICP-AES or flame atomic absorption spectrometer (FAAS); calculating the chemical compositions of coating layer as the content of the analytical element divided by the pre-measured coating mass.

4.2.6 Carbon, C; Silicon, Si; Manganese, Mn; Phosphorus, P; Sulfur, S; Chromium, Cr; Nickel, Ni; Aluminium, Al; Titanium, Ti; Copper, Cu:

Document: ISO 19272:2015, *Low alloyed steel — Determination of C, Si, Mn, P, S, Cr, Ni, Al, Ti and Cu - Glow discharge optical emission spectrometry (routine method).*

Range of application:

- determination of carbon contents from a mass fraction of 0,060 % to a mass fraction of 0,35 %;
- determination of silicon contents from a mass fraction of 0,14 % to a mass fraction of 1,50 %;
- determination of manganese contents from a mass fraction of 0,090 % to a mass fraction of 0,70 %;
- determination of phosphorus contents from a mass fraction of 0,010 % to a mass fraction of 0,070 %;
- determination of sulfur contents from a mass fraction of 0,005 % to a mass fraction of 0,050 %;
- determination of chromium contents from a mass fraction of a mass fraction of 0,008 % to a mass fraction of 0,65 %;
- determination of nickel contents from a mass fraction of 0,050 % to a mass fraction of 0,50 %;
- determination of aluminium contents from a mass fraction of 0,006 % to a mass fraction of 0,90 %;
- determination of titanium contents from a mass fraction of 0,014 % to a mass fraction of 0,13 %;
- determination of copper contents from a mass fraction of 0,005 % to a mass fraction of 1,00 %.

Principle of the method:

a) a sample with a flat and smooth surface is used as the cathode of a direct current or radio frequency glow discharge device;

b) cathodic sputtering of the sample surface, diffusion of the sputtered atoms and ions from the sample surface; excitation of these atoms and ions in the plasma formed in the glow discharge device;

c) spectrometric measurement of the intensity of the emitted light from the ions or the atoms of the elements to be determined, and, optionally, the emitted light from iron at 371,994 nm or 271,441 nm or another appropriate wavelength (if internal standard is used);

d) conversion of the measured signals to the contents through calibration curves established by measuring CRMs.

4.3 General documents

4.3.1 This document, i.e. ISO/TR 9769:—, *Steel and iron — Review of available methods of analysis*.

Scope: facilitates reference to published International Standards for the determination of the chemical composition of steel and iron.

4.3.2 Document: ISO 13898-1:1997, *Steel and iron — Determination of nickel, copper and cobalt contents — Inductively coupled plasma atomic emission spectrometric method — Part 1: General requirements and sample dissolution*.

Principle of the method:

a) specifies the general requirements for analysis by ICP-AES, preparation and dissolution of the test portion and method of calculation and the procedures used for the evaluation of the precision data of the individual methods specified in the subsequent parts;

b) specifies two alternative methods of evaluating concentrations in test solutions; one with and one without the use of an internal standard.

4.3.3 Document: ISO 14284:1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*.

Principle of the method: specifies methods for sampling and sample preparation for the determination of the chemical composition of pig iron, cast iron and steel; methods are specified for use with both liquid and solid metal.

Annex A

(informative)

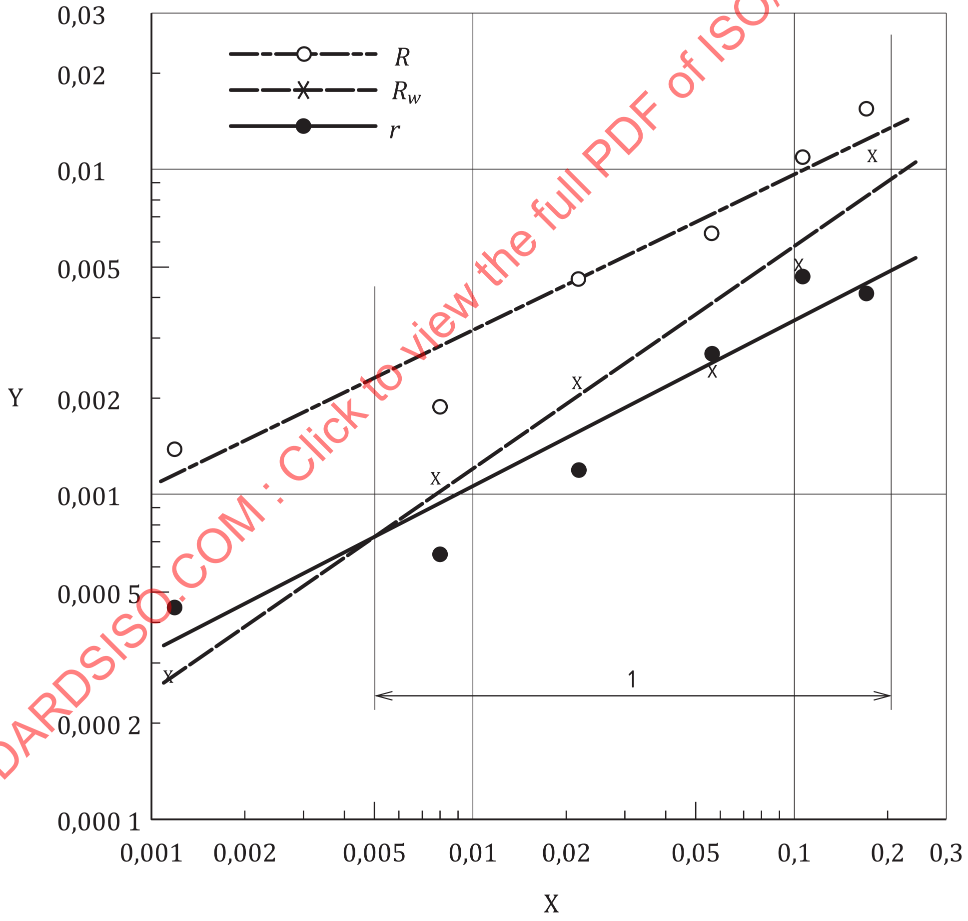
Graphical representation of precision data for the methods presented in this document

A.1 Aluminium, Al

Document: ISO 9658:1990:

— aluminium (acid-soluble): flame atomic absorption spectrometric method, see Figure A.1.

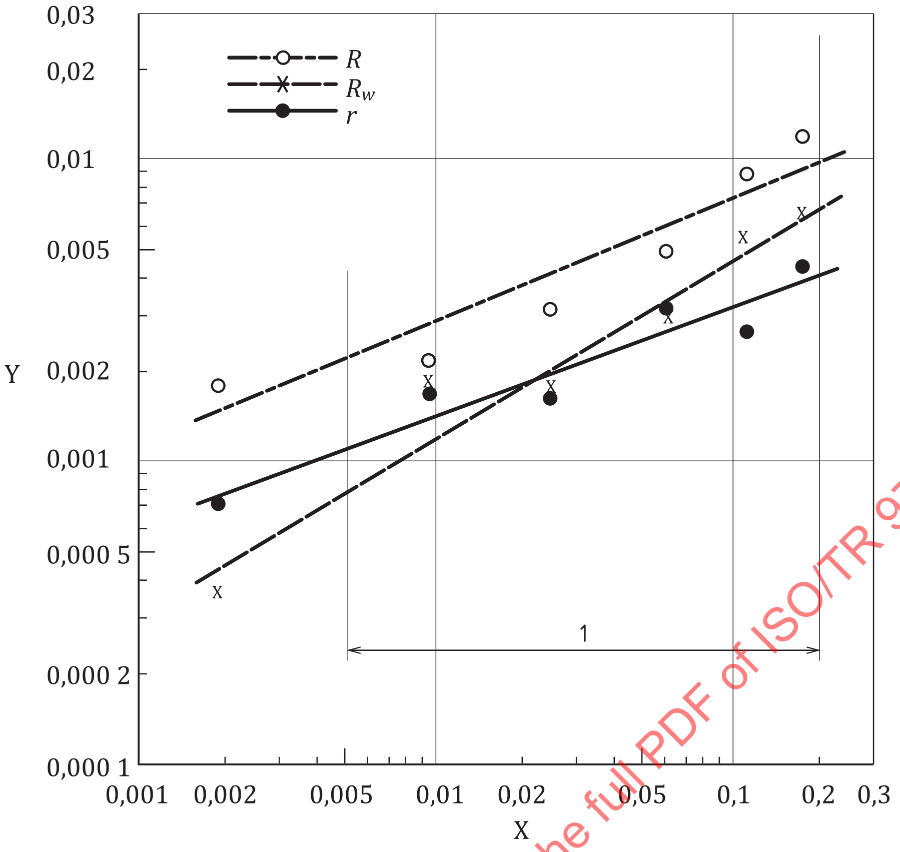
— aluminium (total): flame atomic absorption spectrometric method, see Figure A.2.



Key

X	acid-soluble aluminium content, % (mass fraction)	R	reproducibility
Y	precision, % (mass fraction)	R_w	within-laboratory reproducibility
1	application range	r	repeatability

Figure A.1 — Logarithmic relationship between acid-soluble aluminium content ($\overline{w}_{\text{Al}}$) and repeatability (r) and reproducibility (R and R_W)



Key

X total aluminium content, % (mass fraction)

Y precision, % (mass fraction)

1 application range

R reproducibility

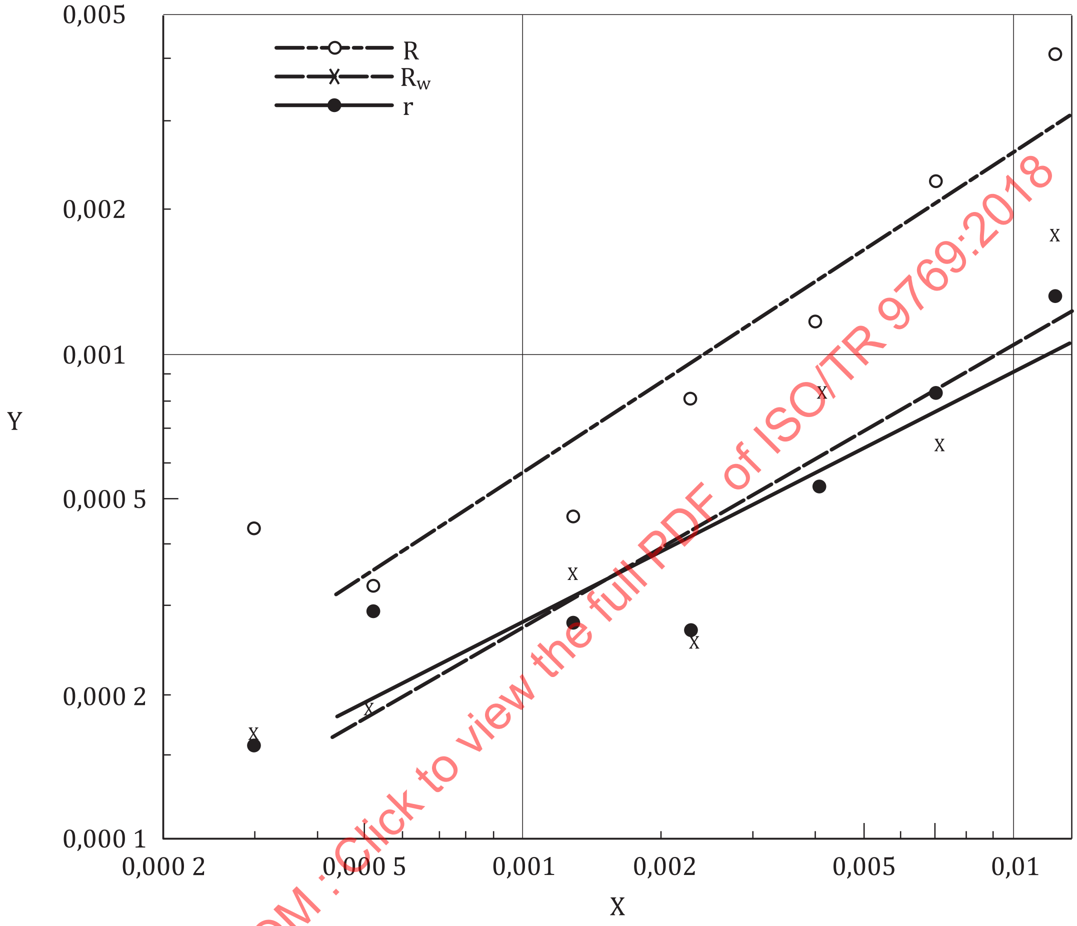
R_w within-laboratory reproducibility

r repeatability

Figure A.2 — Logarithmic relationship between total aluminium content ($\bar{w}_{\mathrm{Al}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.2 Antimony, Sb

Document: ISO 10698:1994, see Figure A.3.



Key

X antimony content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}512\,7 \lg \overline{w}_{\text{Sb},1} - 2{,}008\,7$$

$$\lg R_W = 0{,}592\,2 \lg \overline{w}_{\text{Sb},2} - 1{,}784\,7$$

$$\lg R = 0{,}616\,1 \lg \overline{w}_{\text{Sb},1} - 1{,}461\,8$$

where

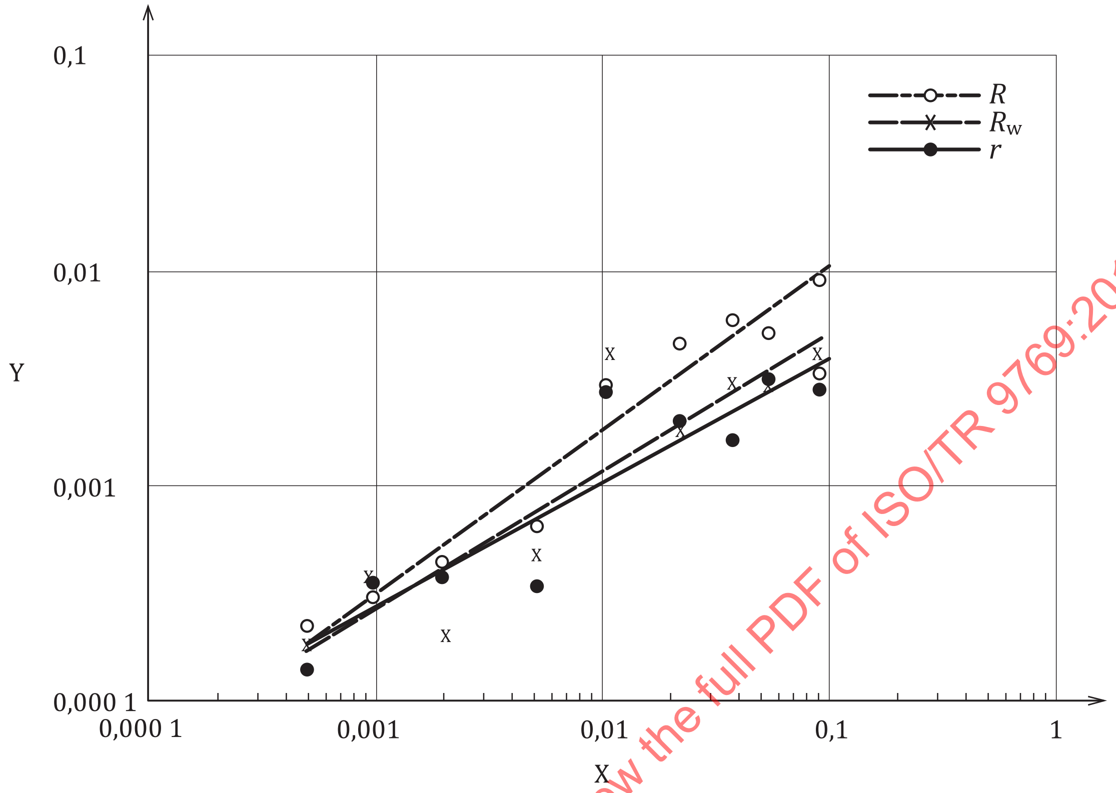
$\overline{w}_{\text{Sb},1}$ is the average antimony content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\text{Sb},2}$ is the average antimony content, expressed as a percentage by mass, obtained between days.

Figure A.3 — Logarithmic relationship between antimony content ($\overline{w}_{\text{Sb}}$) and repeatability (r) and reproducibility (R and R_W)

A.3 Arsenic, As

Document: ISO 17058:2004, see Figure A.4.



Key

X arsenic content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}582\,9 \lg \overline{w}_{\text{As},1} - 1{,}824\,8$$

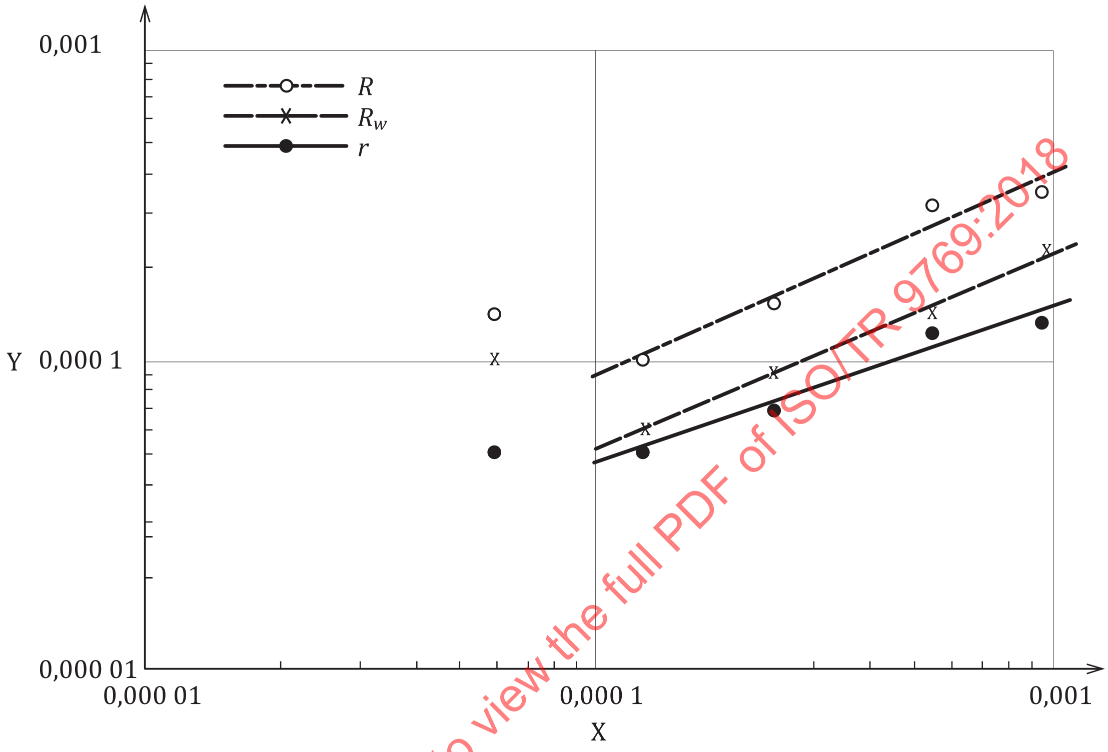
$$\lg R_W = 0{,}65 \lg \overline{w}_{\text{As},2} - 1{,}630\,8$$

$$\lg R = 0{,}765\,4 \lg \overline{w}_{\text{As},1} - 1{,}208\,4$$

Figure A.4 — Logarithmic relationship between arsenic content ($\overline{w}_{\text{As}}$) and repeatability (r) and reproducibility (R and R_W)

A.4 Boron, B

A.4.1 Document: ISO 10153:1997, see Figures A.5 and A.6.



Key

X boron content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}503 \lg \overline{w}_{\mathrm{B},1} - 2{,}346$$

$$\lg R_W = 0{,}625 \lg \overline{w}_{\mathrm{B},2} - 1{,}792$$

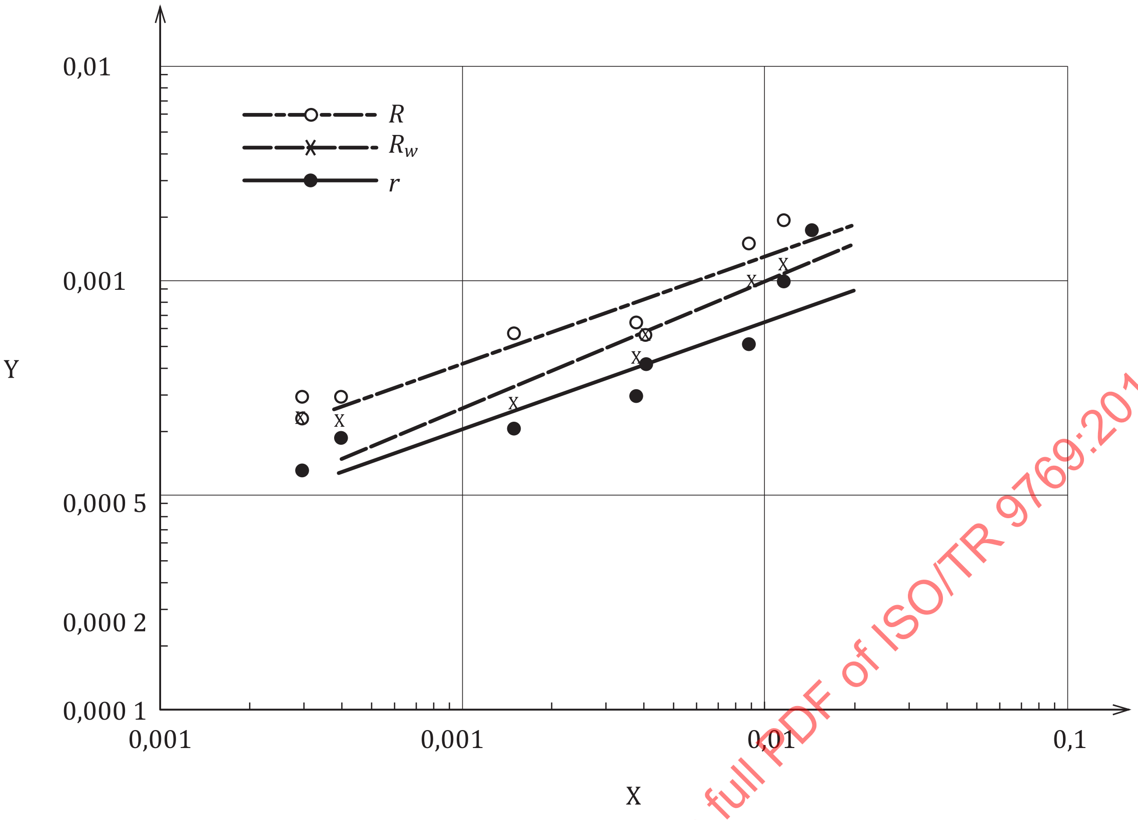
$$\lg R = 0{,}656 \lg \overline{w}_{\mathrm{B},1} - 1{,}446$$

where

$\overline{w}_{\mathrm{B},1}$ is the average boron content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{B},2}$ is the average boron content, expressed as a percentage by mass, obtained between days.

Figure A.5 — Logarithmic relationship between boron content ($\overline{w}_{\mathrm{B}}$) and repeatability ($r$) and reproducibility ($R$ and R_W)



Key

X boron content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}474 \lg \overline{w}_{B,1} - 2{,}275$$

$$\lg R_W = 0{,}563 \lg \overline{w}_{B,2} - 1{,}909$$

$$\lg R = 0{,}501 \lg \overline{w}_{B,1} - 1{,}898$$

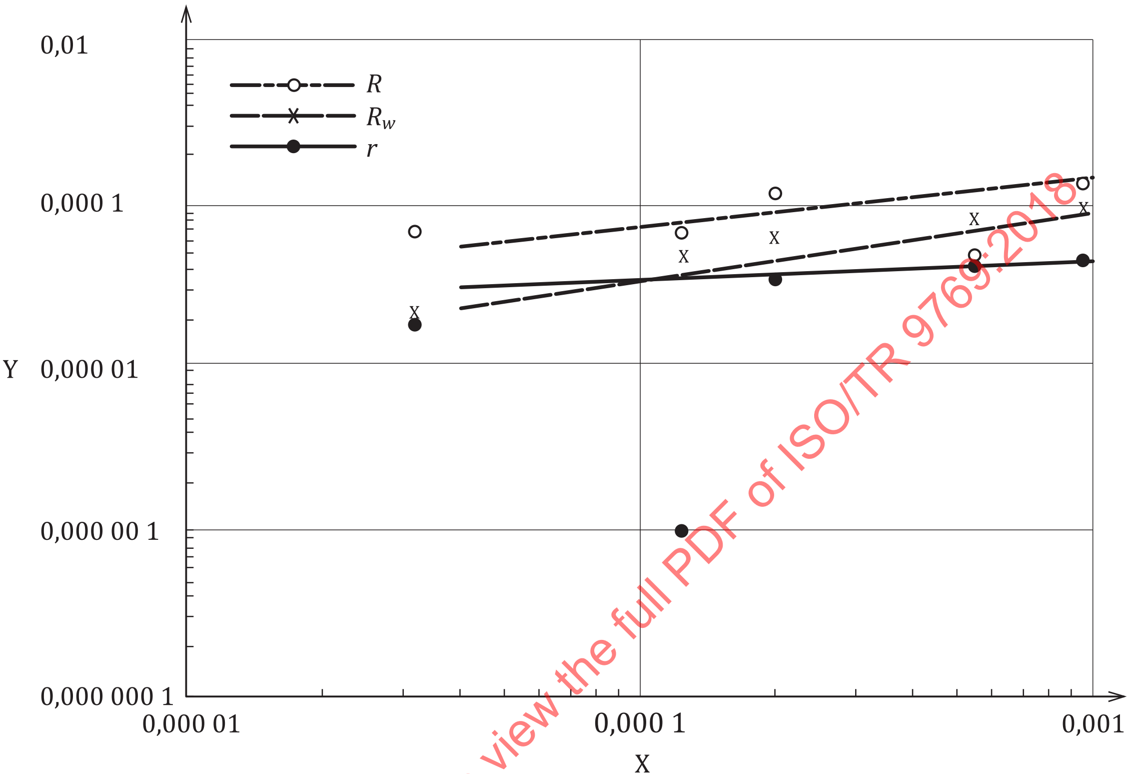
where

$\overline{w}_{B,1}$ is the average boron content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{B,2}$ is the average boron content, expressed as a percentage by mass, obtained between days.

Figure A.6 — Logarithmic relationship between boron content ($\overline{w}_B$) and repeatability limit (r) and reproducibility limits (R and R_W)

A.4.2 Document: ISO 13900:1997, see Figure A.7.



Key

X boron content, % (mass fraction)
Y precision, % (mass fraction)
R reproducibility
R_W within-laboratory reproducibility
r repeatability

$$\lg r = 0{,}144\,1 \lg \overline{w}_{\mathrm{B},1} + 0{,}249$$

$$\lg R_W = 0{,}341 \lg \overline{w}_{\mathrm{B},2} - 2{,}943$$

$$\lg R = 0{,}130 \lg \overline{w}_{\mathrm{B},1} - 3{,}605$$

where

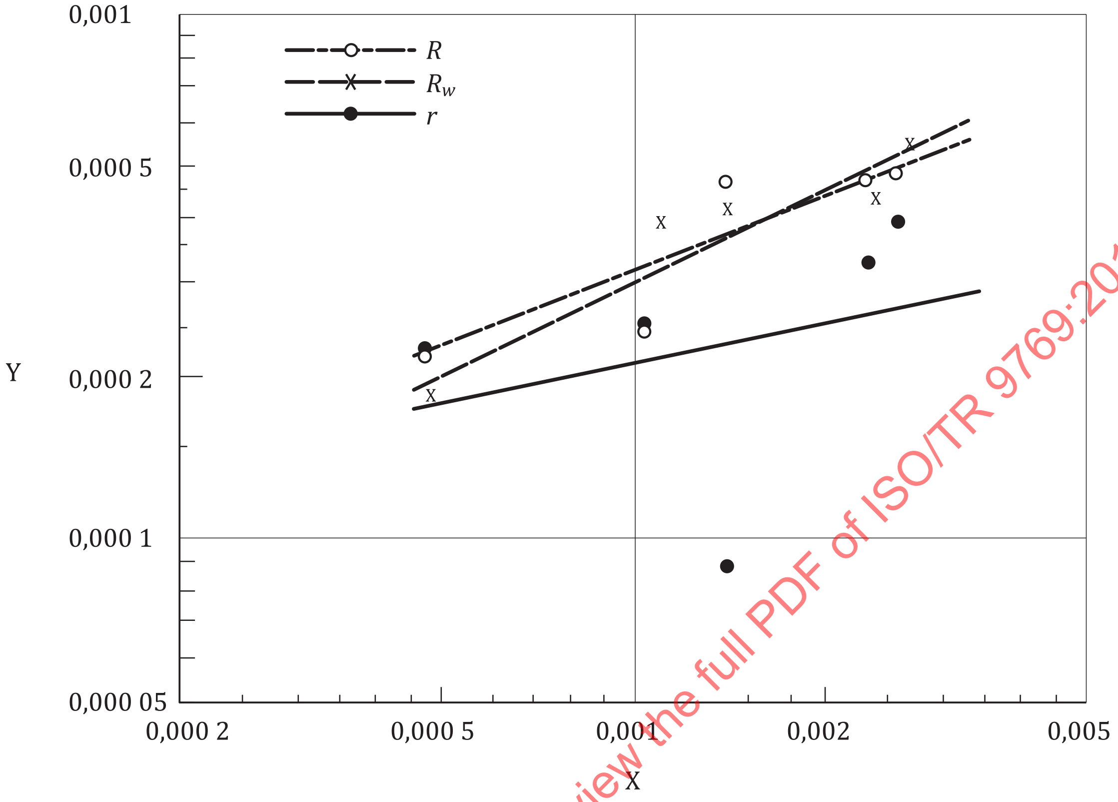
$\overline{w}_{\mathrm{B},1}$ is the average boron content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{B},2}$ is the average boron content, expressed as a percentage by mass, obtained between days.

Figure A.7 — Logarithmic relationship between boron content ($\overline{w}_{\mathrm{B}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.5 Calcium, Ca

A.5.1 Document: ISO 10697-1:1992, see Figure A.8.



Key

X	acid-soluble calcium content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

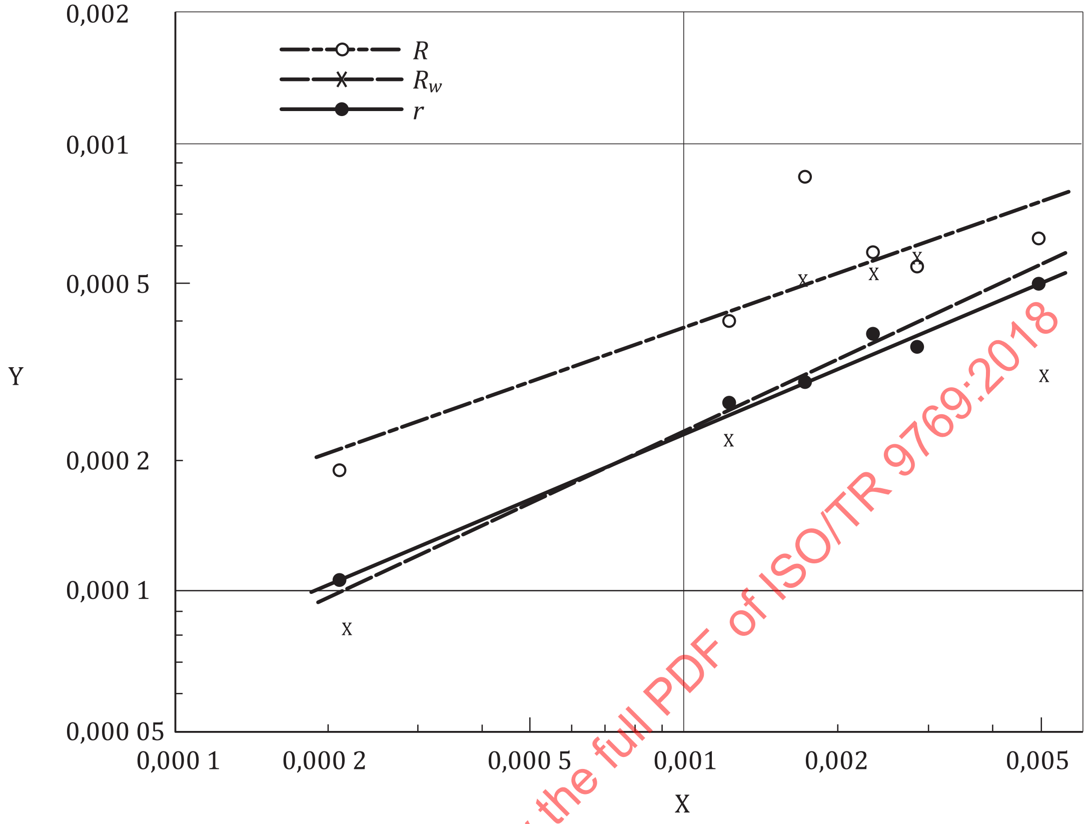
$$\lg r = 0{,}297\,2 \lg \overline{w}_{\mathrm{Ca},1} - 2{,}806\,0$$

$$\lg R_W = 0{,}646\,1 \lg \overline{w}_{\mathrm{Ca},2} - 1{,}573\,4$$

$$\lg R = 0{,}256\,3 \lg \overline{w}_{\mathrm{Ca},1} - 2{,}665\,8$$

Figure A.8 — Logarithmic relationship between acid-soluble calcium content ($\overline{w}_{\mathrm{Ca}}$) and repeatability ($r$) and reproducibility ($R$ and R_W)

A.5.2 Document: ISO 10697-2:1994, see Figure A.9.



Key

X	calcium content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

$$\lg r = 0{,}478\,5 \lg \bar{w}_{\mathrm{Ca},1} - 2{,}188\,8$$

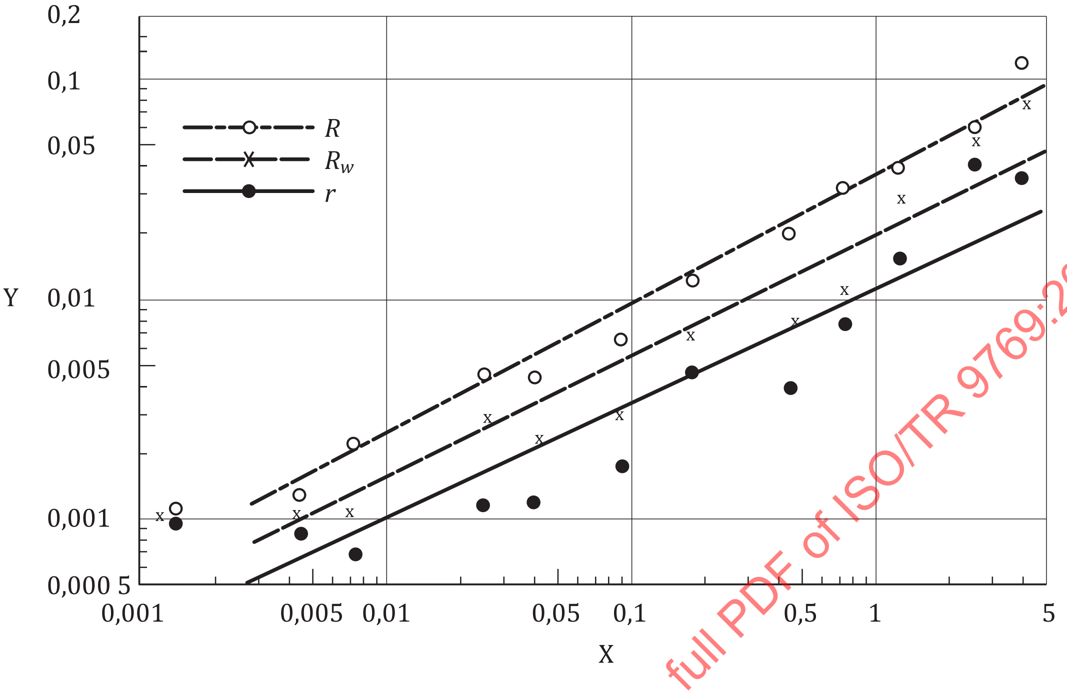
$$\lg R_W = 0{,}540\,4 \lg \bar{w}_{\mathrm{Ca},2} - 2{,}005\,3$$

$$\lg R = 0{,}408\,7 \lg \bar{w}_{\mathrm{Ca},1} - 2{,}166\,7$$

Figure A.9 — Logarithmic relationship between total calcium content ($\bar{w}_{\mathrm{Ca}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.6 Carbon, C

A.6.1 Document: ISO 9556:1989, see Figure A.10.

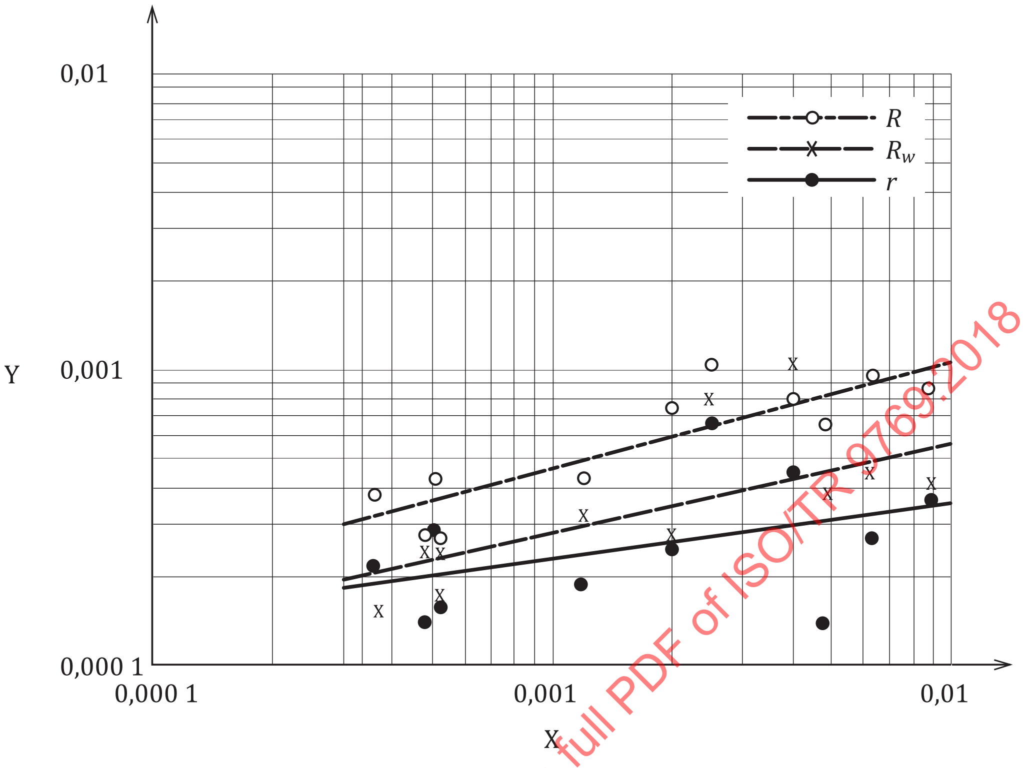


Key

X	carbon content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

Figure A.10 — Logarithmic relationship between carbon content ($\bar{w}_C$) and repeatability (*r*) and reproducibility (R_w and *R*)

A.6.2 Document: ISO 15349-2:1999, see Figure A.11.



Key

X	carbon content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

$$\lg r = 0{,}185\,6 \lg \overline{w}_{\mathrm{C},1} - 3{,}087\,1$$

$$\lg R_W = 0{,}326\,8 \lg \overline{w}_{\mathrm{C},2} - 2{,}574\,2$$

$$\lg R = 0{,}367\,9 \lg \overline{w}_{\mathrm{C},1} - 2{,}231\,2$$

where

$\overline{w}_{\mathrm{C},1}$ is the average carbon content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{C},2}$ is the average carbon content, expressed as a percentage by mass, obtained between days.

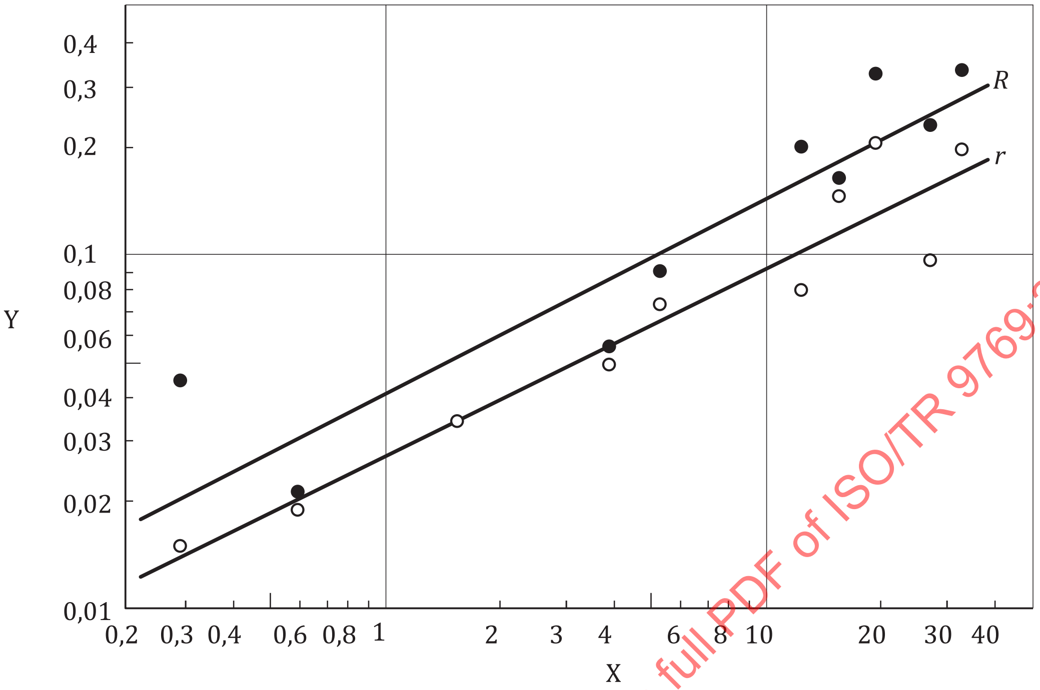
Figure A.11 — Logarithmic relationship between carbon content ($\overline{w}_{\mathrm{C}}$) and repeatability (*r*) and reproducibility (R_w and *R*)

A.6.3 Document: ISO/TS 10719:2016, see Figure A.12.

There is no graphical representation of precision data in the standard.

A.7 Chromium, Cr

A.7.1 Document: ISO 4937:1986, see Figures A.12 and A.13.



Key

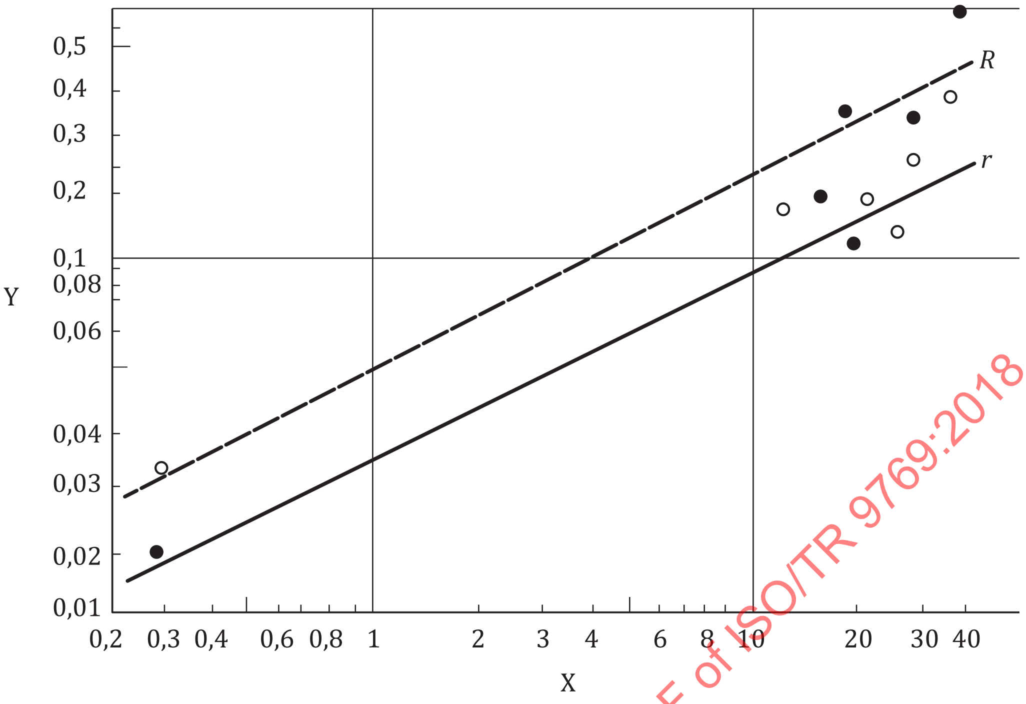
X chromium content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

r repeatability

Figure A.12 — Logarithmic relationship between chromium content ($\bar{w}_{Cr}$) and repeatability (r) and reproducibility (R) — Potentiometric titration



Key

X chromium content, % (mass fraction)

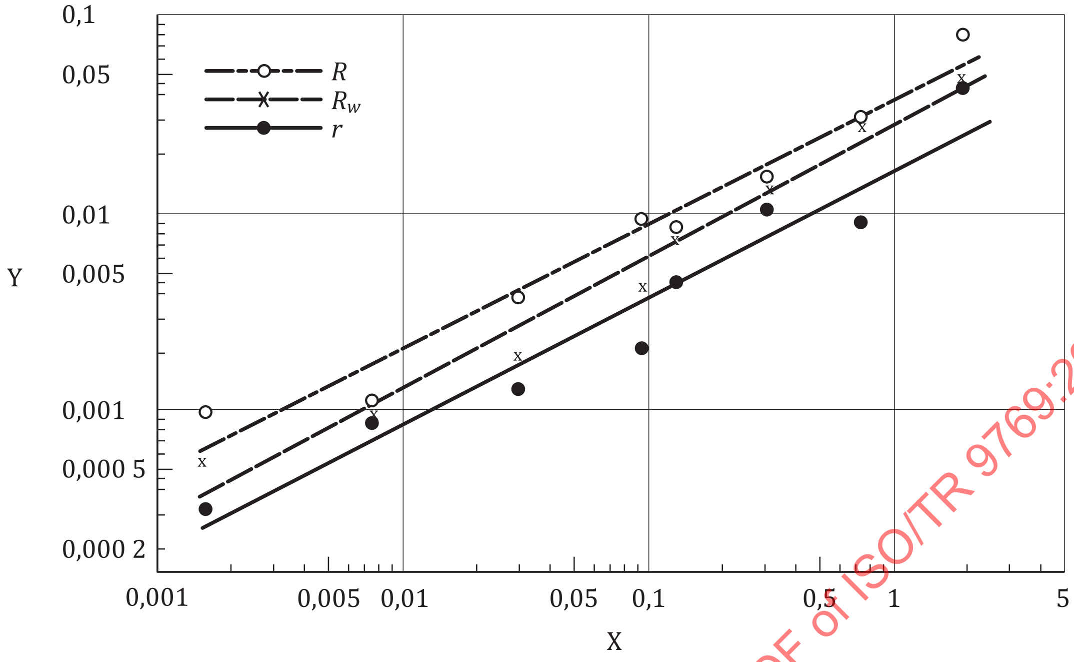
Y precision, % (mass fraction)

R reproducibility

r repeatability

Figure A.13 — Logarithmic relationship between chromium content ($\bar{w}_{\text{Cr}}$) and repeatability (*r*) and reproducibility (*R*) — Visual titration

A.7.2 Document: ISO 10138:1991, see Figure A.14.



Key

X	chromium content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

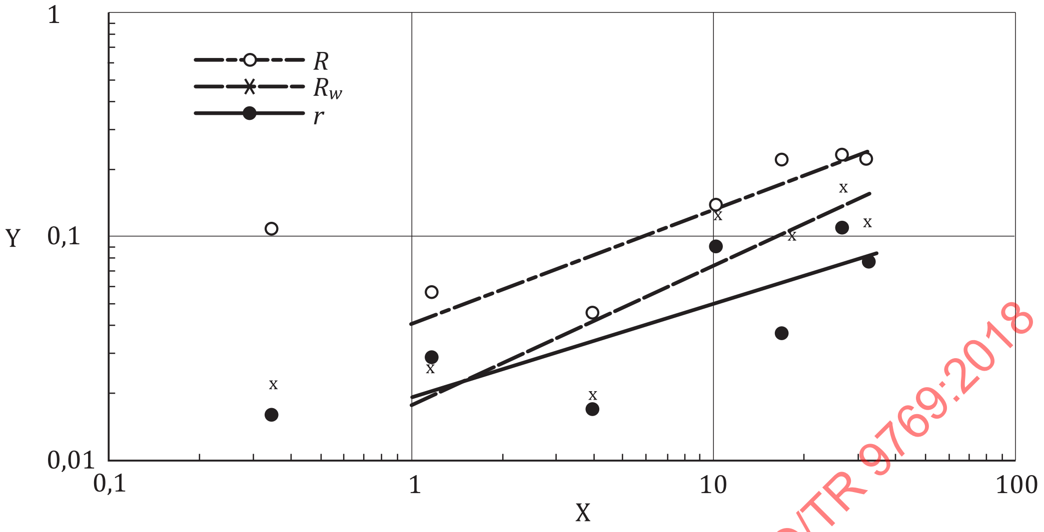
$$\lg r = 0{,}642\ 78 \lg \overline{w}_{\mathrm{Cr}} - 1{,}793\ 4$$

$$\lg R_W = 0{,}667\ 99 \lg \overline{w}_{\mathrm{Cr}} - 1{,}556\ 6$$

$$\lg R = 0{,}639\ 8 \lg \overline{w}_{\mathrm{Cr}} - 1{,}429\ 6$$

Figure A.14 — Logarithmic relationship between chromium content ($\overline{w}_{\mathrm{Cr}}$) and repeatability ($r$) and reproducibility ($R$ and R_W)

A.7.3 Document: ISO 15355:1999, see Figure A.15.



Key

X chromium content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

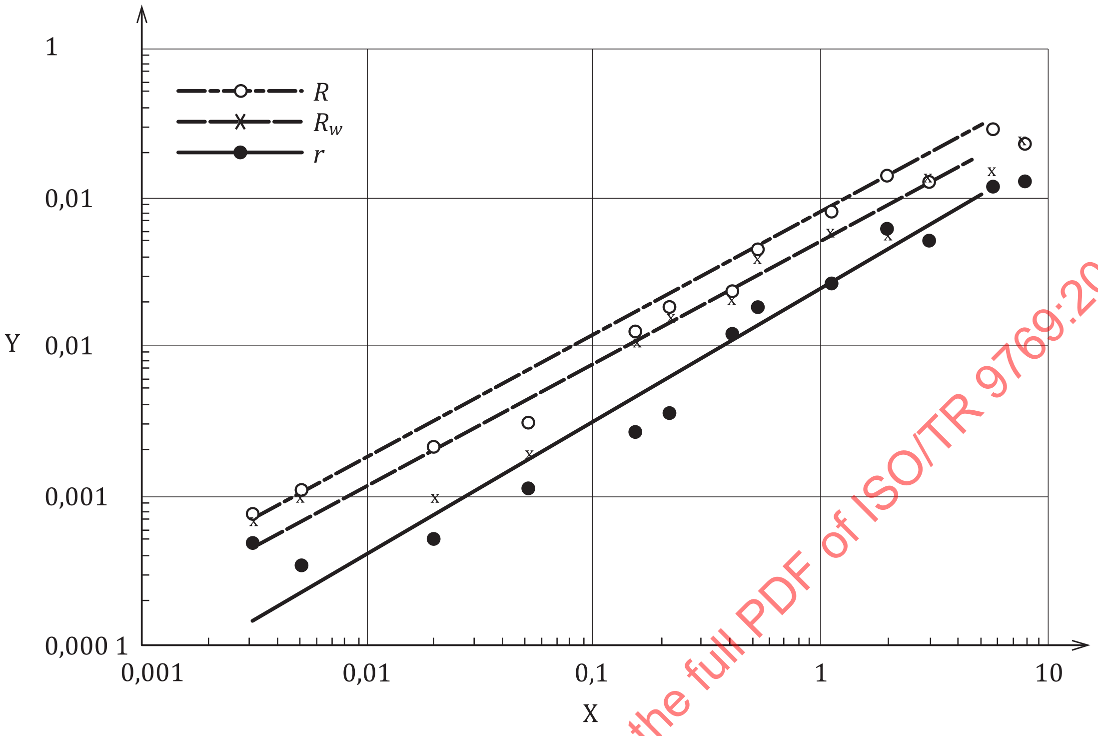
R_W within-laboratory reproducibility

r repeatability

Figure A.15 — Logarithmic relationship between chromium content ($\bar{w}_{\mathrm{Cr}}$) and repeatability (*r*) and reproducibility (R_W and *R*)

A.8 Cobalt, Co

A.8.1 Document: ISO 11652:1997, see Figure A.16.



Key

X cobalt content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}820\,2 \lg \overline{w}_{\text{Co},1} - 1{,}663\,8$$

$$\lg R_W = 0{,}787\,3 \lg \overline{w}_{\text{Co},2} - 1{,}374\,9$$

$$\lg R = 0{,}790\,0 \lg \overline{w}_{\text{Co},1} - 1{,}243\,0$$

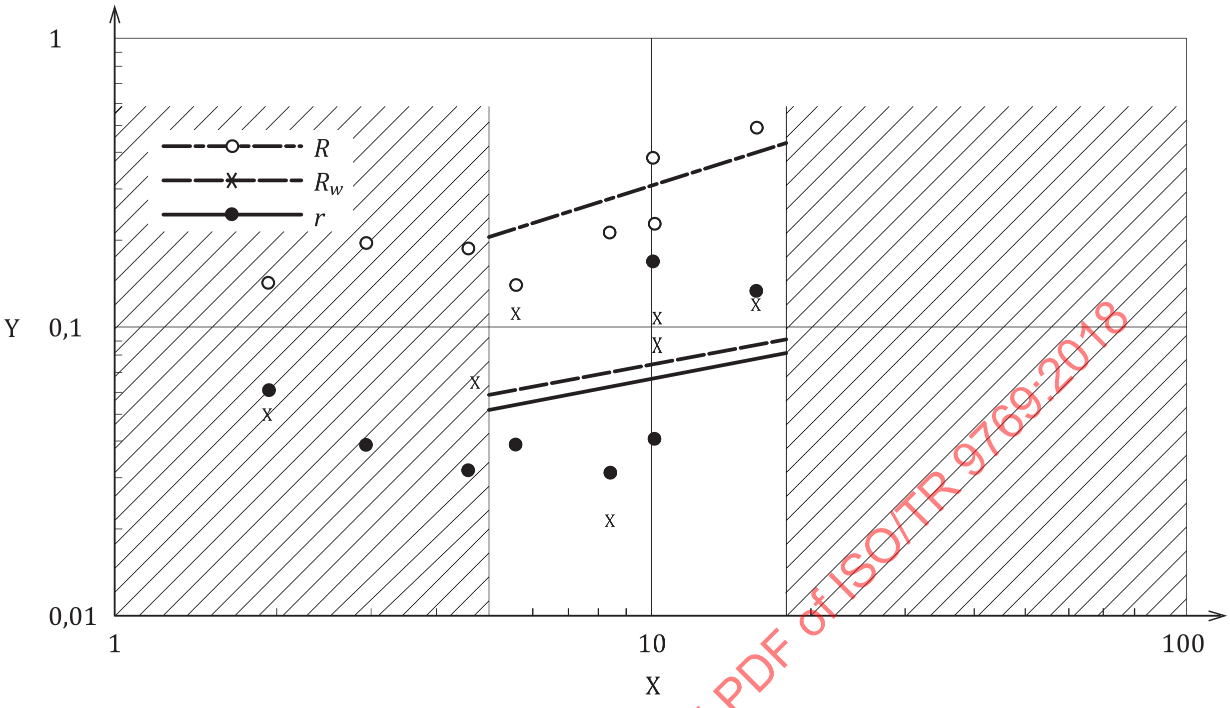
where

$\overline{w}_{\text{Co},1}$ is the average cobalt content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\text{Co},2}$ is the average cobalt content, expressed as a percentage by mass, obtained between days.

Figure A.16 — Logarithmic relationship between cobalt content ($\overline{w}_{\text{Co}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.8.2 Document: ISO 11653:1997, see Figure A.17.



Key

X cobalt content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}403\,0 \lg \overline{w}_{\mathrm{Co},1} - 1{,}577$$

$$\lg R_W = 0{,}399\,1 \lg \overline{w}_{\mathrm{Co},2} - 1{,}500$$

$$\lg R = 0{,}493\,4 \lg \overline{w}_{\mathrm{Co},1} - 1{,}039$$

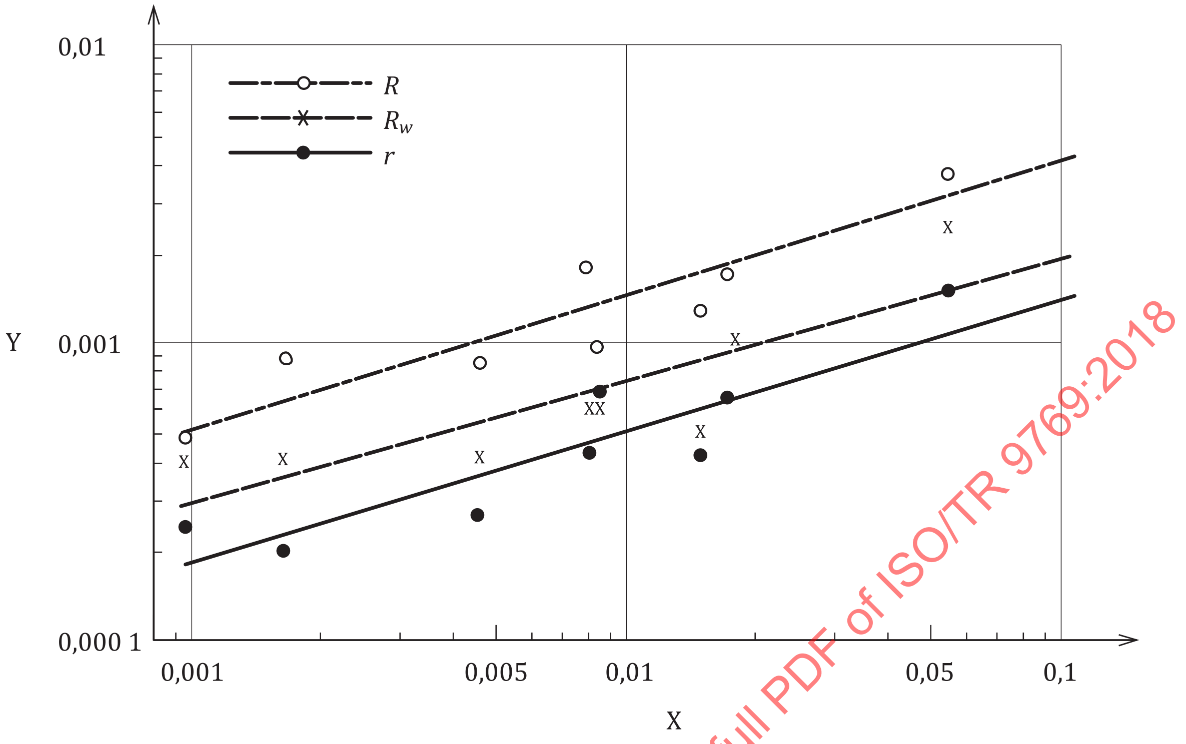
where

$\overline{w}_{\mathrm{Co},1}$ is the average cobalt content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{Co},2}$ is the average cobalt content, expressed as a percentage by mass, obtained between days.

Figure A.17 — Logarithmic relationship between cobalt content ($\overline{w}_{\mathrm{Co}}$) and repeatability ($r$) and reproducibility ($R$ and R_W)

A.8.3 Document: ISO 13898-4:1997, see Figure A.18.



Key

X cobalt content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}439\,5 \lg \overline{w}_{\text{Co},1} - 2{,}421\,1$$

$$\lg R_W = 0{,}392\,8 \lg \overline{w}_{\text{Co},2} - 2{,}340\,9$$

$$\lg R = 0{,}445\,9 \lg \overline{w}_{\text{Co},1} - 1{,}953\,8$$

where

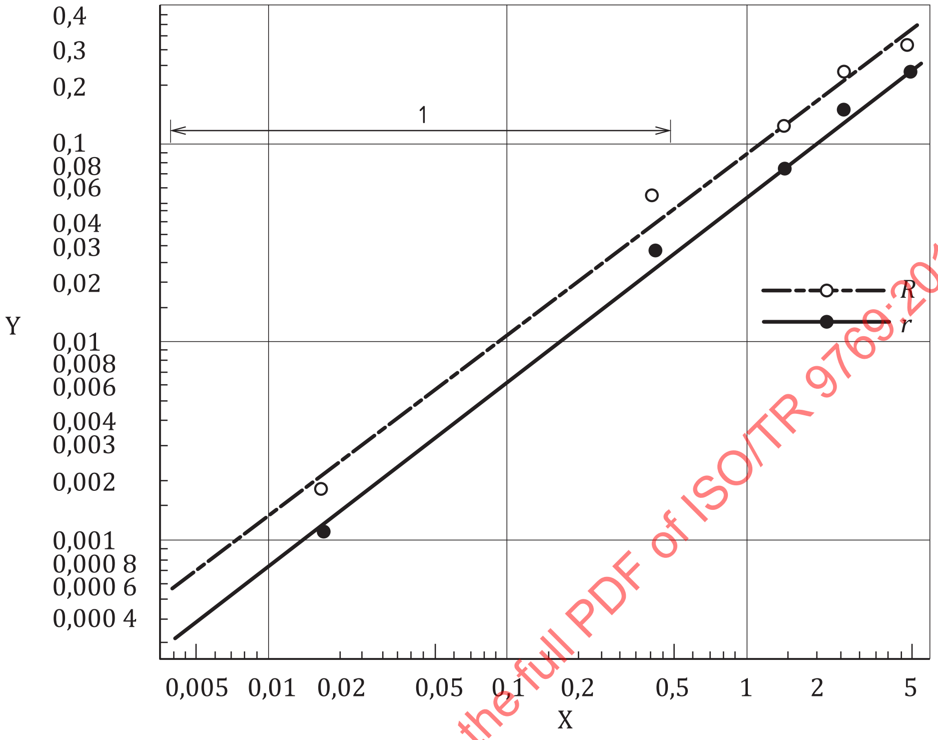
$\overline{w}_{\text{Co},1}$ is the average cobalt content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\text{Co},2}$ is the average cobalt content, expressed as a percentage by mass, obtained between days.

Figure A.18 — Logarithmic relationship between cobalt content ($\overline{w}_{\text{Co}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.9 Copper, Cu

A.9.1 Document: ISO 4943:1985, see Figure A.19.

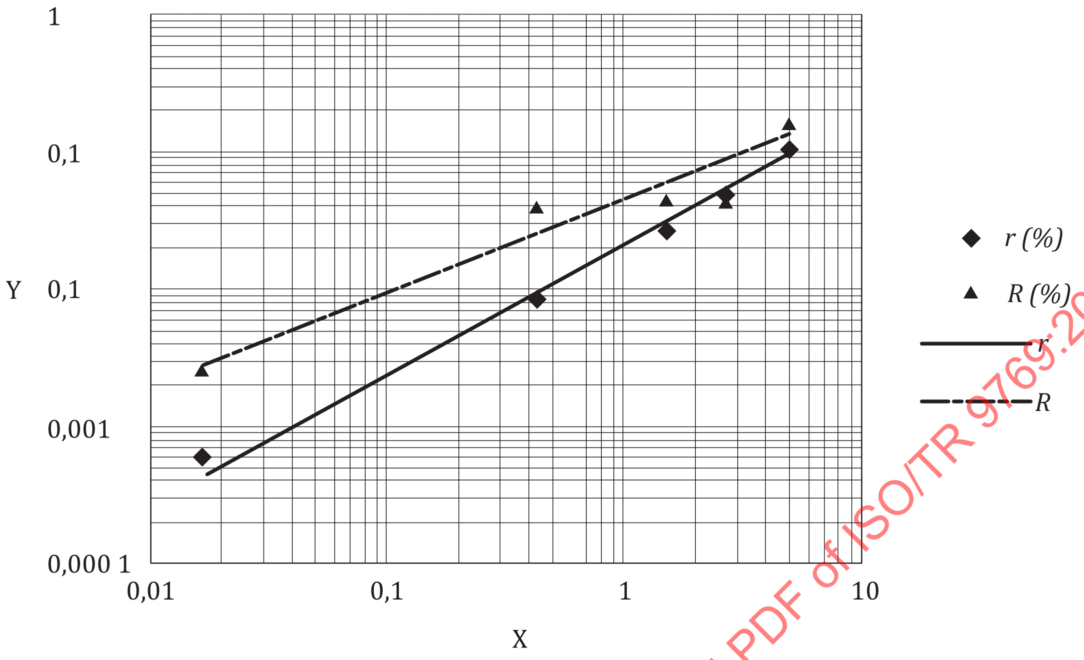


Key

X	copper content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
r	repeatability
1	application range

Figure A.19 — Logarithmic relationship between copper content ($\bar{w}_{Cu}$) and repeatability (*r*) and reproducibility (*R*)

A.9.2 Document: ISO 4946:2016, see Figure A.20.



Key

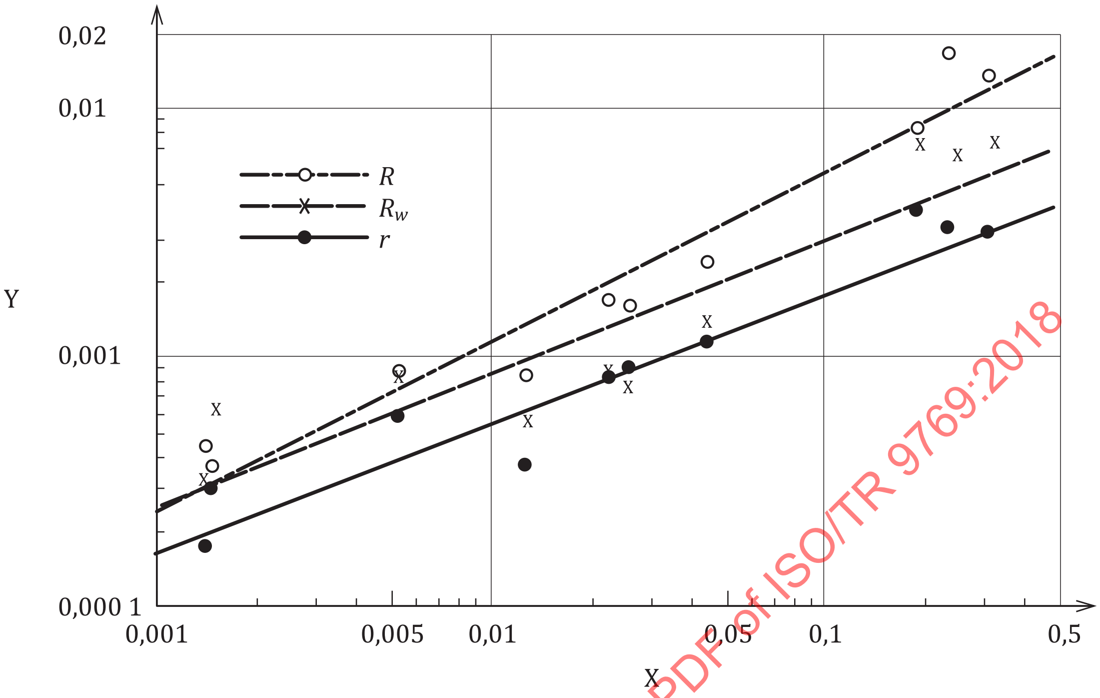
X	copper content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
r	repeatability

$$\lg r = 0{,}941\,3 \lg \bar{w}_{\text{Cu}} - 1{,}679$$

$$\lg R = 0{,}680\,2 \lg \bar{w}_{\text{Cu}} - 1{,}339$$

Figure A.20 — Logarithmic relationship between copper content ($\bar{w}_{\text{Cu}}$) and repeatability (r) and reproducibility (R)

A.9.3 Document: ISO 13898-3:1997, see Figure A.21.



Key

X	copper content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

$$\lg r = 0{,}530\,9 \lg \overline{w}_{\text{Cu},1} - 2{,}209\,4$$

$$\lg R_W = 0{,}537\,4 \lg \overline{w}_{\text{Cu},2} - 2{,}000\,3$$

$$\lg R = 0{,}681\,6 \lg \overline{w}_{\text{Cu},1} - 1{,}588\,8$$

where

$\overline{w}_{\text{Cu},1}$ is the average copper content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\text{Cu},2}$ is the average copper content, expressed as a percentage by mass, obtained between days.

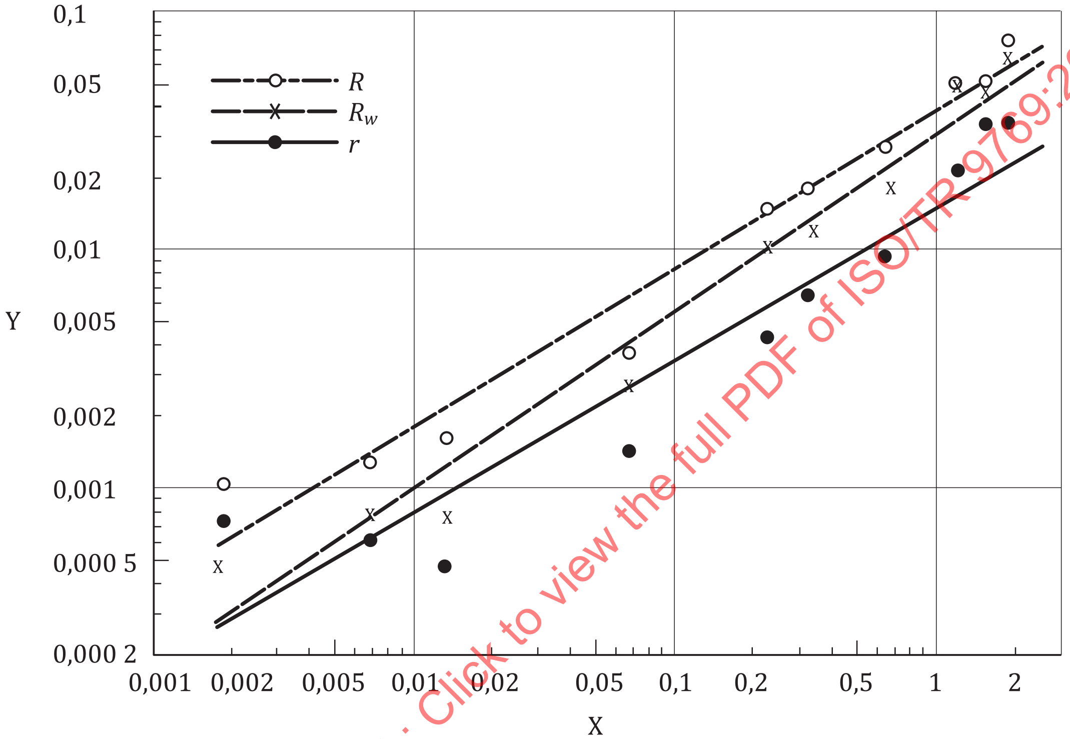
Figure A.21 — Logarithmic relationship between copper content ($\overline{w}_{\text{Cu}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.10 Manganese, Mn

A.10.1 Document: ISO 629:1982.

There is no graphical representation of precision data in the standard.

A.10.2 Document: ISO 10700:1994, see Figure A.22.



Key

X	manganese content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

$$\lg r = 0{,}641\,8 \lg \overline{w}_{\text{Mn},1} - 1{,}806\,3$$

$$\lg R_\text{W} = 0{,}742\,0 \lg \overline{w}_{\text{Mn},2} - 1{,}519\,5$$

$$\lg R = 0{,}661\,7 \lg \overline{w}_{\text{Mn},1} - 1{,}415\,7$$

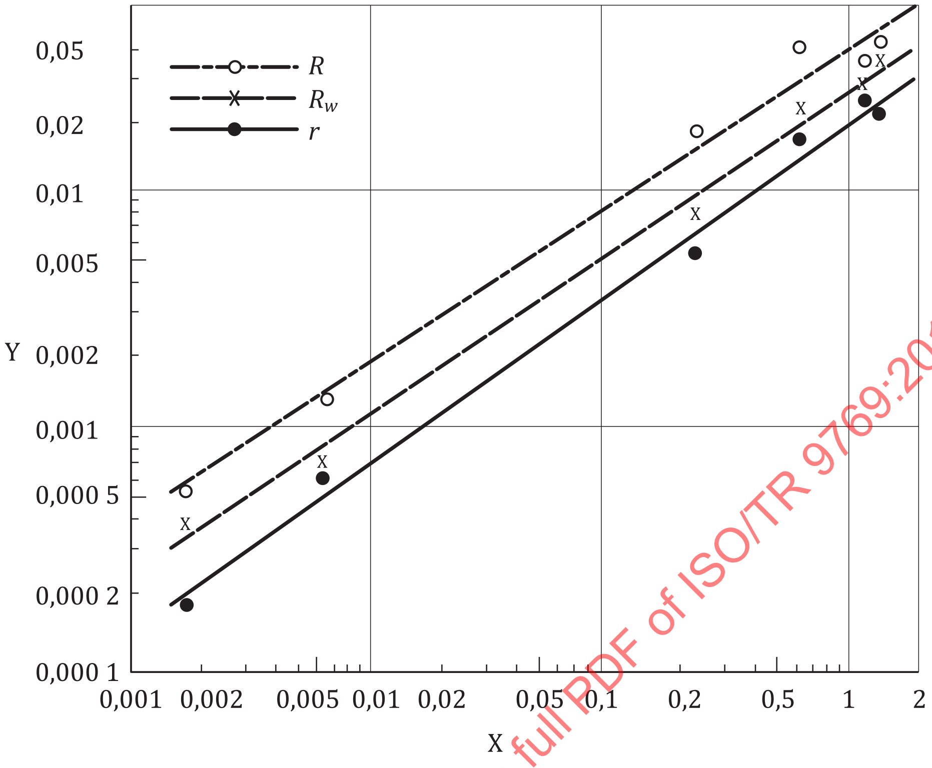
where

$\overline{w}_{\text{Mn},1}$ is the average manganese content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\text{Mn},2}$ is the average manganese content, expressed as a percentage by mass, obtained between days.

Figure A.22 — Logarithmic relationship between manganese content ($\overline{w}_{\text{Mn}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.10.3 Document: ISO 10278:1995, see Figure A.23.



Key

X	manganese content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

$$\lg r = 0{,}718\,6 \lg \overline{w}_{\mathrm{Mn},1} - 1{,}701\,2$$

$$\lg R_{\mathrm{W}} = 0{,}676\,8 \lg \overline{w}_{\mathrm{Mn},2} - 1{,}591\,5$$

$$\lg R = 0{,}668\,6 \lg \overline{w}_{\mathrm{Mn},1} - 1{,}389\,1$$

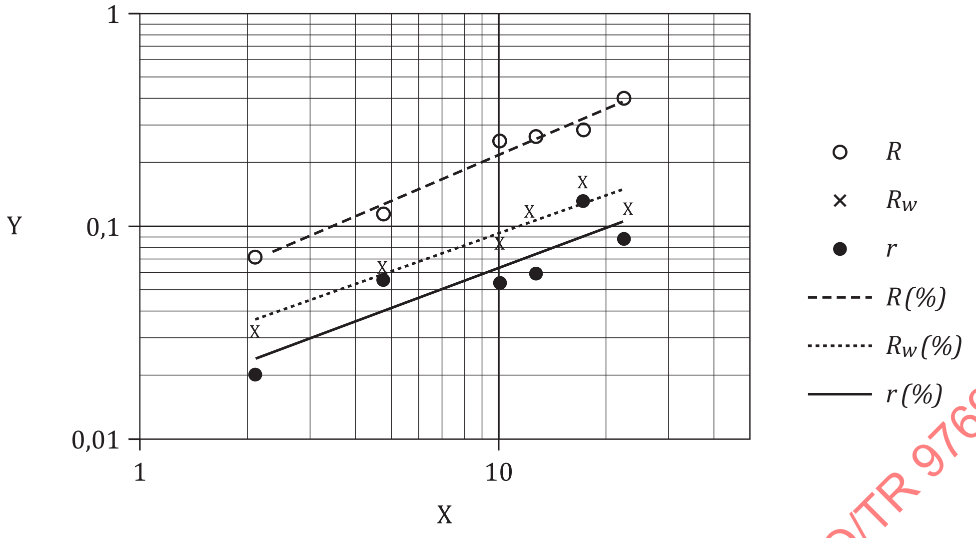
where

$\overline{w}_{\mathrm{Mn},1}$ is the average manganese content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{Mn},2}$ is the average manganese content, expressed as a percentage by mass, obtained between days.

Figure A.23 — Logarithmic relationship between manganese content ($\overline{w}_{\mathrm{Mn}}$) and repeatability (*r*) and reproducibility (*R* and R_{W})

A.10.4 Document: ISO 18632:2018, see Figure A.24.



Key

X manganese content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}627\,4 \lg \bar{w}_{\text{Mn}} - 1{,}823\,9$$

$$\lg R_W = 0{,}594\,7 \lg \bar{w}_{\text{Mn}} - 1{,}627\,1$$

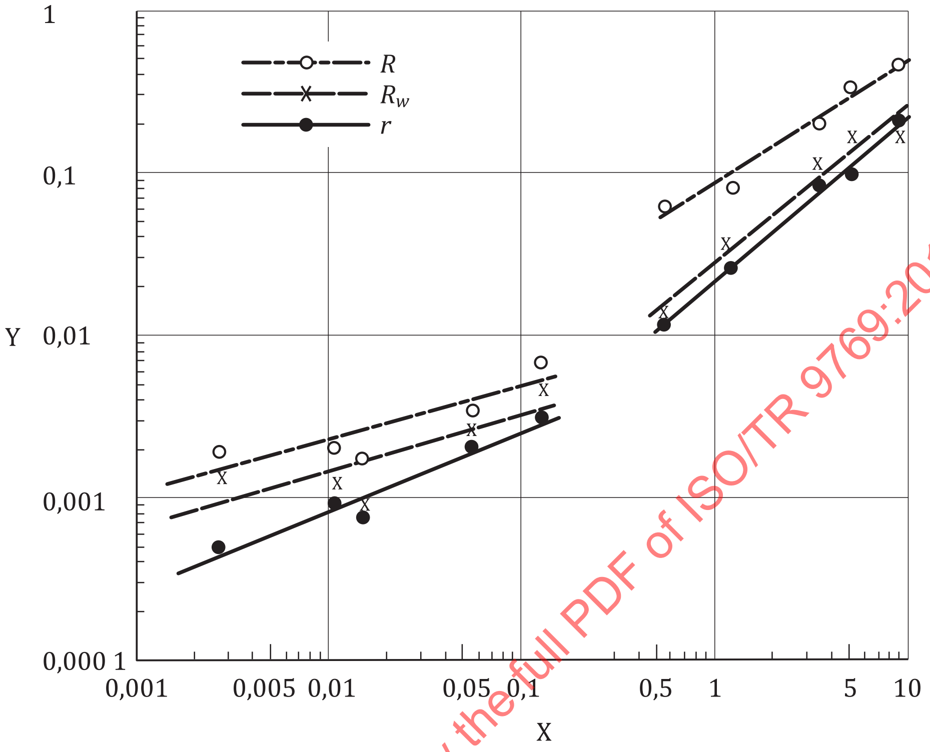
$$\lg R = 0{,}721\,3 \lg \bar{w}_{\text{Mn}} - 1{,}386\,2$$

where $\bar{w}_{\text{Mn}}$ is the average manganese content, expressed as a percentage by mass, obtained from three determinations in each laboratory.

Figure A.24 — Logarithmic relationship between manganese content ($\bar{w}_{\text{Mn}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.11 Molybdenum, Mo

A.11.1 Document: ISO 4941:1994, see Figure A.25.



Key

X molybdenum content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}485\,2 \lg \overline{w}_{\text{Mo},1} - 2{,}115$$

$$\lg R_W = 0{,}327\,9 \lg \overline{w}_{\text{Mo},2} - 2{,}172$$

$$\lg R = 0{,}309\,0 \lg \overline{w}_{\text{Mo},1} - 2{,}025$$

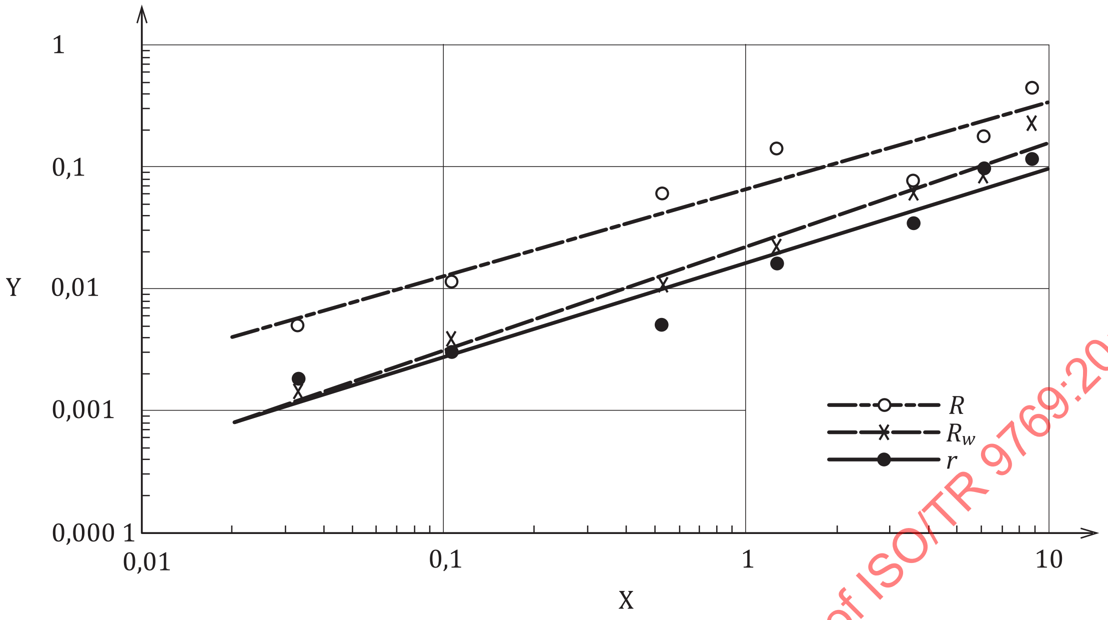
where

$\overline{w}_{\text{Mo},1}$ is the average molybdenum content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\text{Mo},2}$ is the average molybdenum content, expressed as a percentage by mass, obtained between days.

Figure A.25 — Logarithmic relationship between molybdenum content ($\overline{w}_{\text{Mo}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.11.2 Document: ISO/TS 13899-1:2004, see Figure A.26.



Key

X molybdenum content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}777\,6 \lg \overline{w}_{\text{Mo}} - 1{,}788\,1$$

$$\lg R_W = 0{,}844\,5 \lg \overline{w}_{\text{Mo}} - 1{,}652\,2$$

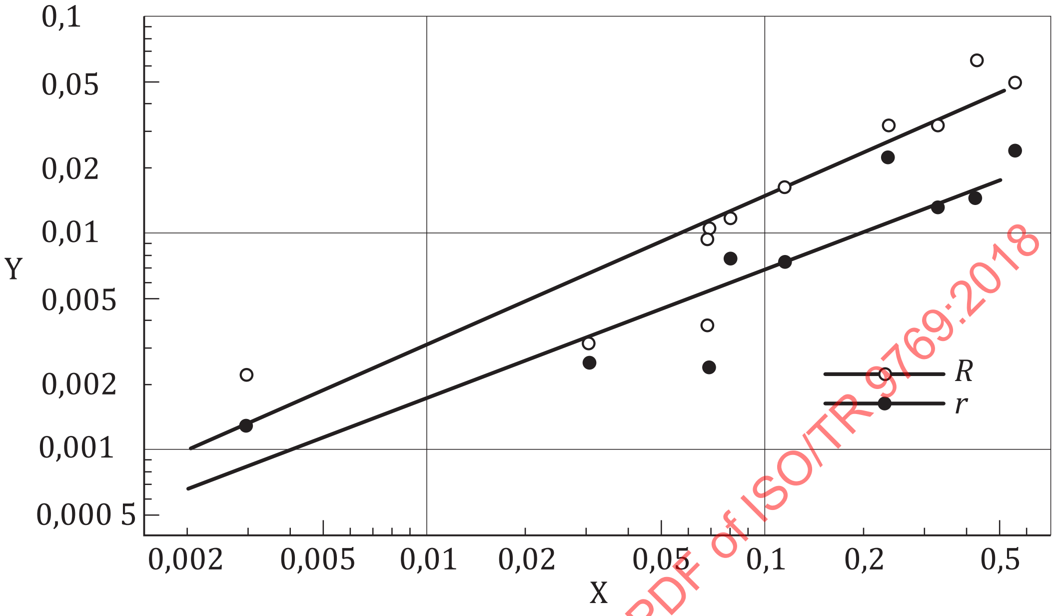
$$\lg R = 0{,}712\,4 \lg \overline{w}_{\text{Mo}} - 1{,}176\,1$$

where $\overline{w}_{\text{Mo}}$ is the average molybdenum content, expressed as a percentage by mass, obtained from three determinations in each laboratory.

Figure A.26 — Logarithmic relationship between molybdenum content ($\overline{w}_{\text{Mo}}$) and repeatability (*r*) and reproducibility (*R* and R_{W})

A.12 Nickel, Ni

A.12.1 Document: ISO 4940:1985, see Figure A.27.



Key

X nickel content, % (mass fraction)

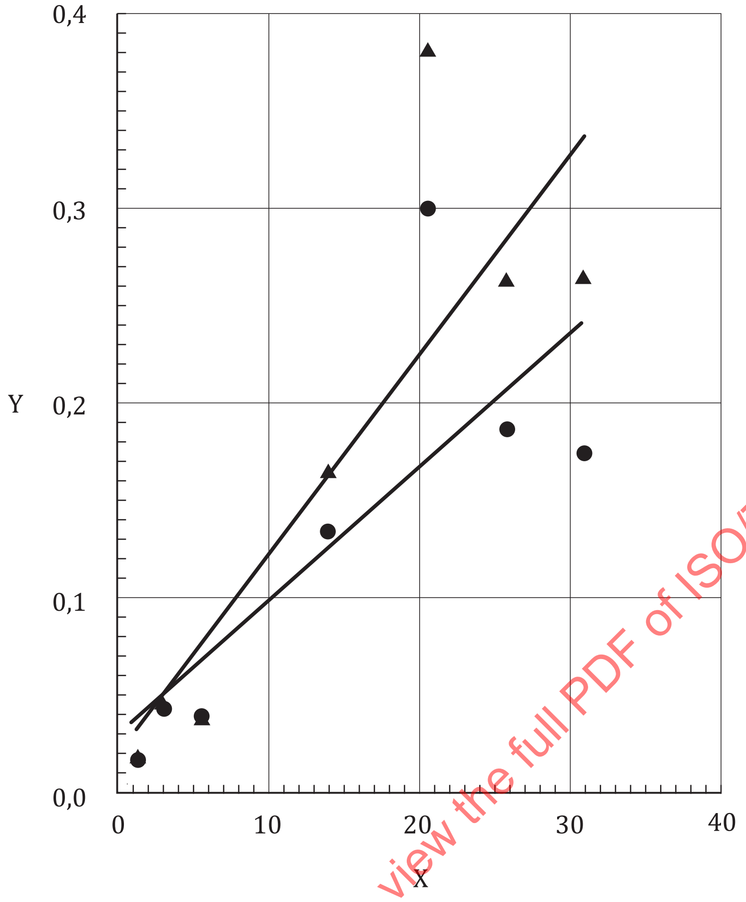
Y precision, % (mass fraction)

R reproducibility

r repeatability

Figure A.27 — Logarithmic relationship between nickel content ($\bar{w}_{\text{Ni}}$) and repeatability (*r*) and reproducibility (*R*)

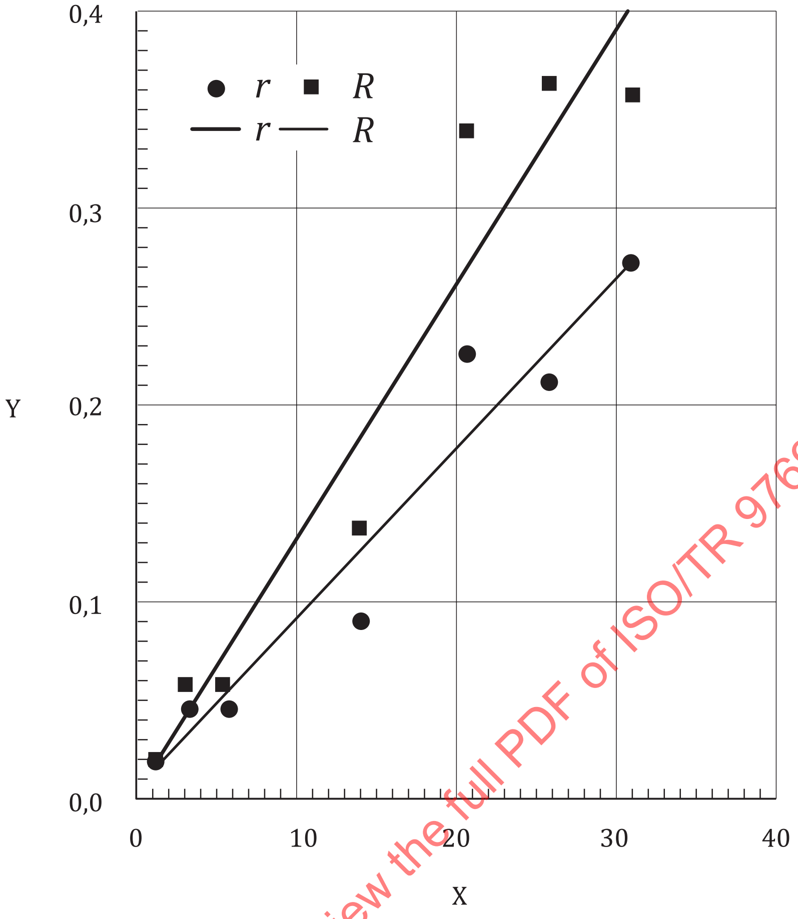
A.12.2 Document: ISO 4938:2016, see Figures A.28 and A.29.



Key

X	nickel content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
r	repeatability

Figure A.28 — Logarithmic relationship between nickel content ($\bar{w}_{\mathrm{Ni}}$) and repeatability (*r*) and reproducibility (*R*) — Gravimetric determination

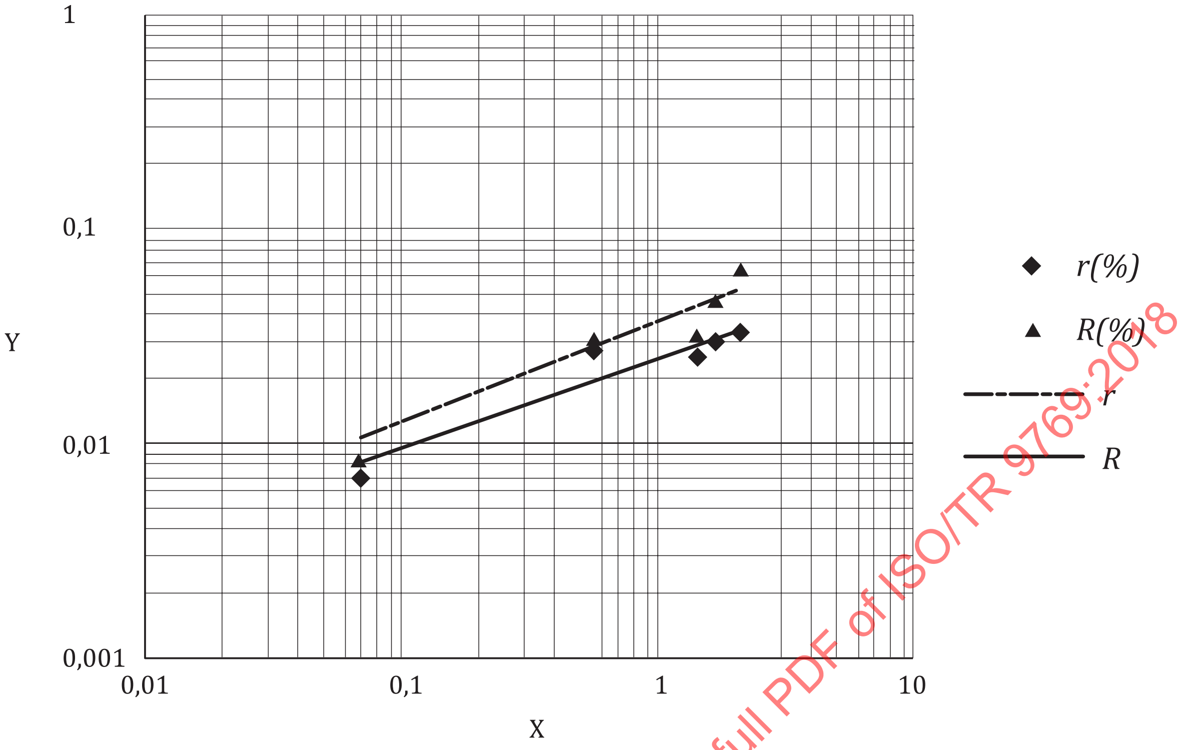


Key

X nickel content, % (mass fraction)
Y precision, % (mass fraction)
R reproducibility
r repeatability

Figure A.29 — Logarithmic relationship between nickel content ($\bar{w}_{Ni}$) and repeatability (*r*) and reproducibility (*R*) — Titrimetric determination

A.12.3 Document: ISO 4939:2016, see Figure A.30.



Key

X nickel content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

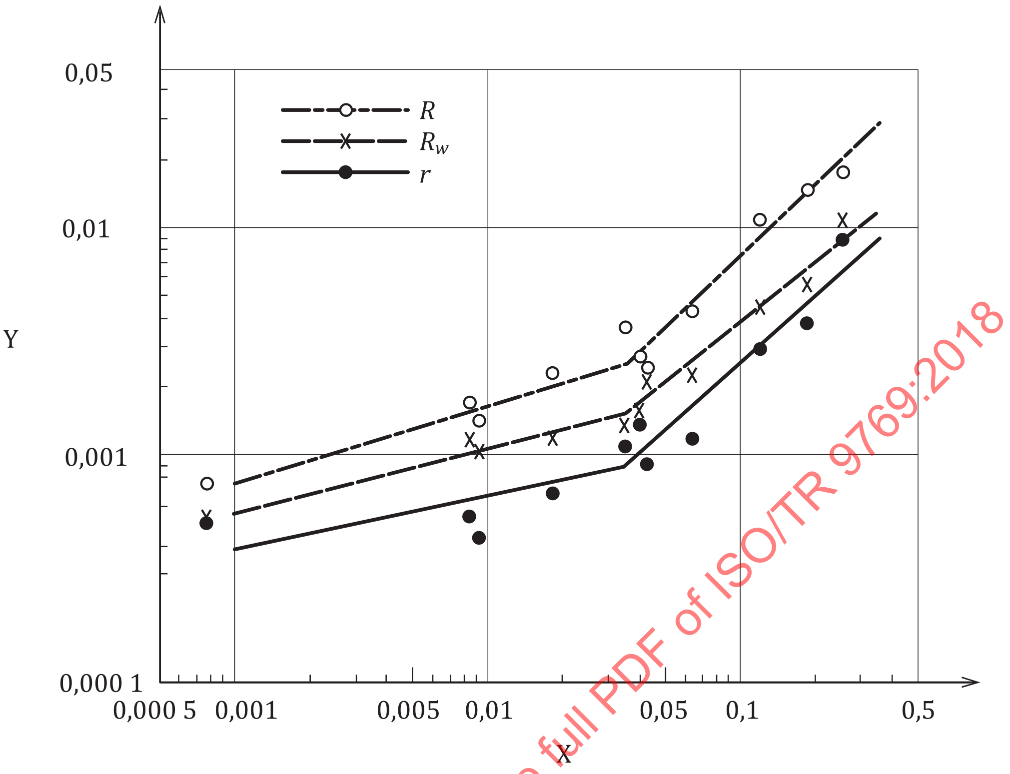
r repeatability

$$\lg r = 0{,}407\,9 \lg \overline{w}_{\mathrm{Ni}} - 1{,}597$$

$$\lg R = 0{,}467\,7 \lg \overline{w}_{\mathrm{Ni}} - 1{,}420$$

Figure A.30 — Logarithmic relationship between nickel content ($\overline{w}_{\mathrm{Ni}}$) and repeatability (*r*) and reproducibility (*R*)

A.12.4 Document: ISO 13898-2:1997, see Figure A.31.



Key

X nickel content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

nickel content up to 0,03 % (mass fraction)

$$\lg r = 0{,}224\,6 \lg \overline{w}_{\mathrm{Ni},1} - 2{,}731\,8$$

$$\lg R_W = 0{,}279\,1 \lg \overline{w}_{\mathrm{Ni},2} - 2{,}419\,0$$

$$\lg R = 0{,}331\,3 \lg \overline{w}_{\mathrm{Ni},1} - 2{,}127\,4$$

nickel content between 0,03 % (mass fraction) and 0,3 % (mass fraction)

$$\lg r = 0{,}994\,1 \lg \overline{w}_{\mathrm{Ni},1} - 1{,}599\,2$$

$$\lg R_W = 0{,}920\,8 \lg \overline{w}_{\mathrm{Ni},2} - 1{,}510\,3$$

$$\lg R = 0{,}988\,8 \lg \overline{w}_{\mathrm{Ni},1} - 1{,}144\,1$$

where

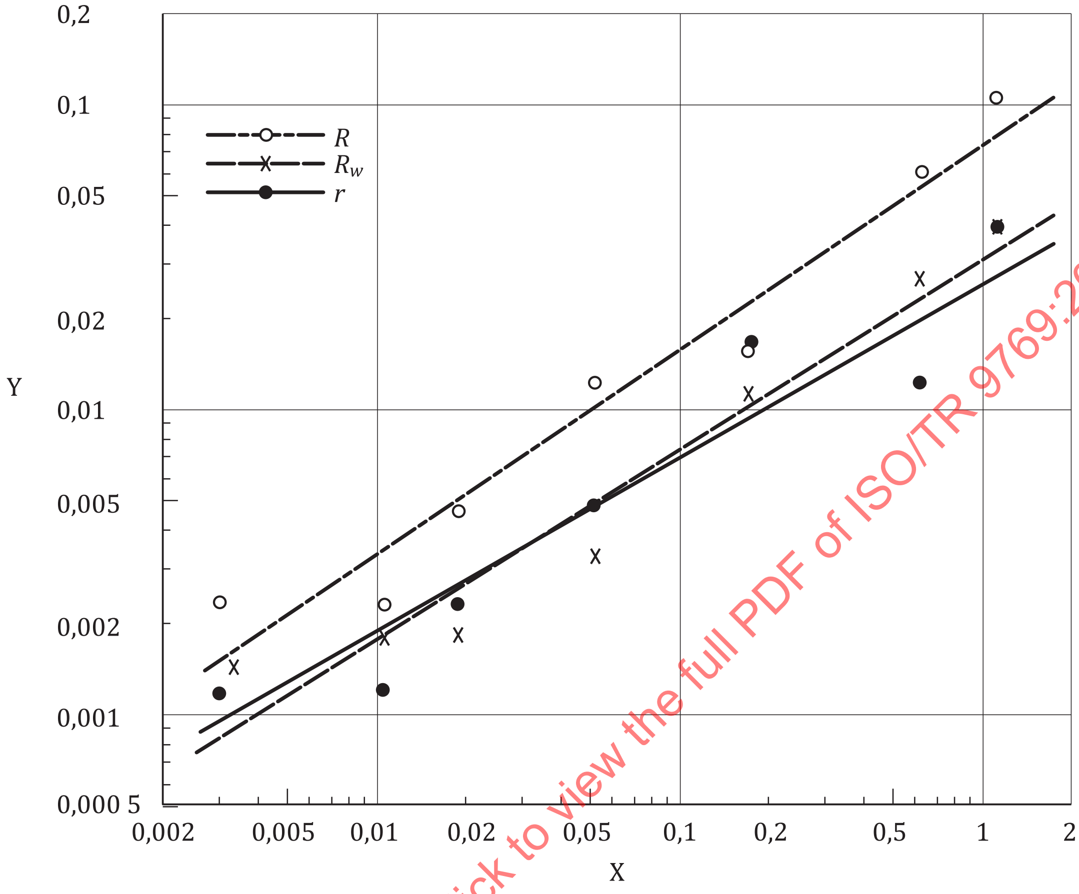
$\overline{w}_{\mathrm{Ni},1}$ is the average nickel content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{Ni},2}$ is the average nickel content, expressed as a percentage by mass, obtained between days.

Figure A.31 — Logarithmic relationship between nickel content ($\overline{w}_{\mathrm{Ni}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.13 Niobium, Nb

A.13.1 Document: ISO 9441:1988, see Figure A.32.



Key

X niobium content, % (mass fraction)

Y precision, % (mass fraction)

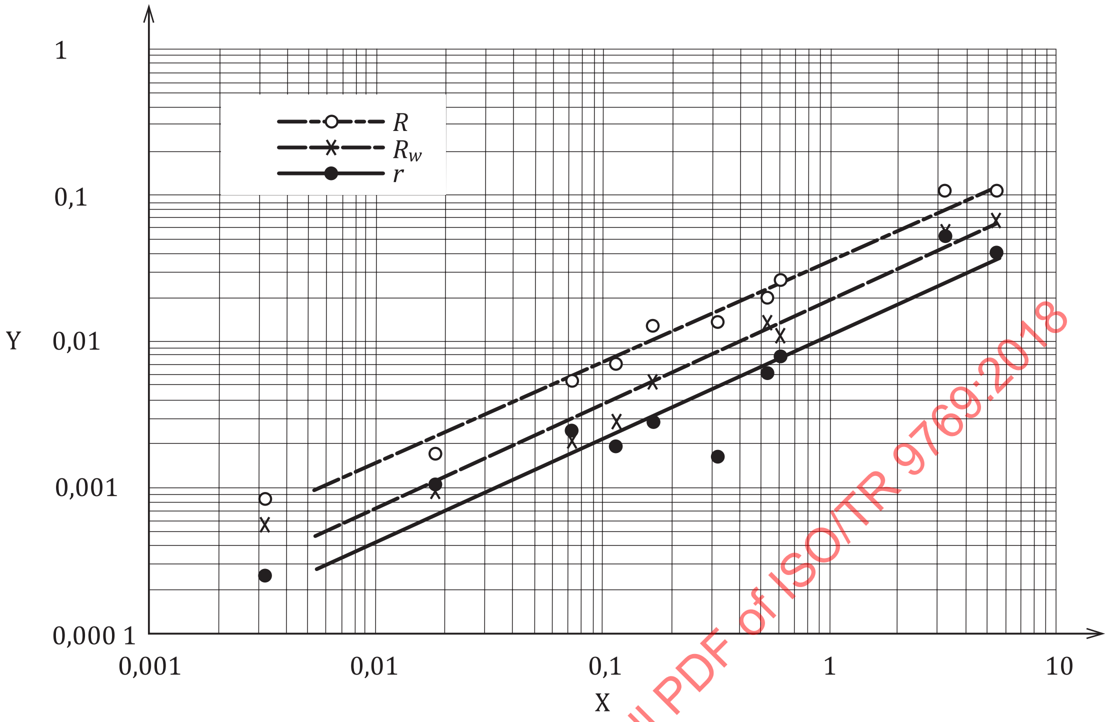
R reproducibility

R_W within-laboratory reproducibility

r repeatability

Figure A.32 — Logarithmic relationship between niobium content ($\overline{w}_{\mathrm{Nb}}$) and repeatability ($r$) and reproducibility ($R$ and R_W)

A.13.2 Document: ISO 13899-2:2005, see Figure A.33.



Key

X	molybdenum content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

$$\lg r = 0{,}689\,6 \lg \overline{w}_{\text{Nb}} - 1{,}903\,4$$

$$\lg R_W = 0{,}701\,4 \lg \overline{w}_{\text{Nb}} - 1{,}655\,3$$

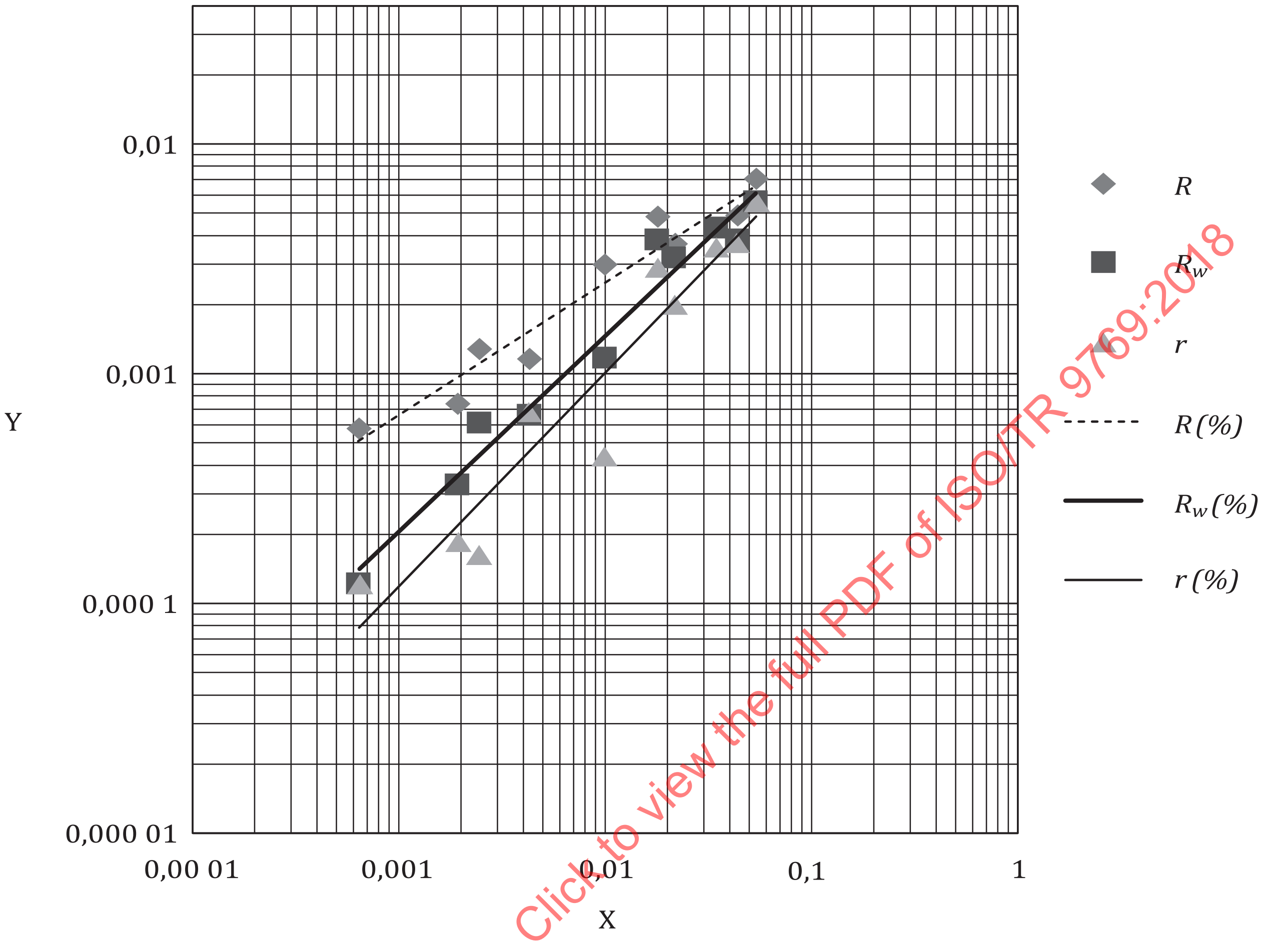
$$\lg R = 0{,}696\,6 \lg \overline{w}_{\text{Nb}} - 1{,}398\,4$$

where $\overline{w}_{\text{Nb}}$ is the average niobium content, expressed as a percentage by mass, obtained from three determinations in each laboratory.

Figure A.33 — Logarithmic relationship between niobium content ($\overline{w}_{\text{Nb}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.14 Nitrogen, N

A.14.1 Document: ISO 4945:2018, see Figure A.34.



Key

X nitrogen content, % (mass fraction)

Y precision, % (mass fraction)

Key

X nitrogen content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$\lg r = 0{,}931\,0 \lg \bar{w}_N - 1{,}133\,7$ Correlation coefficient = 0,956 7

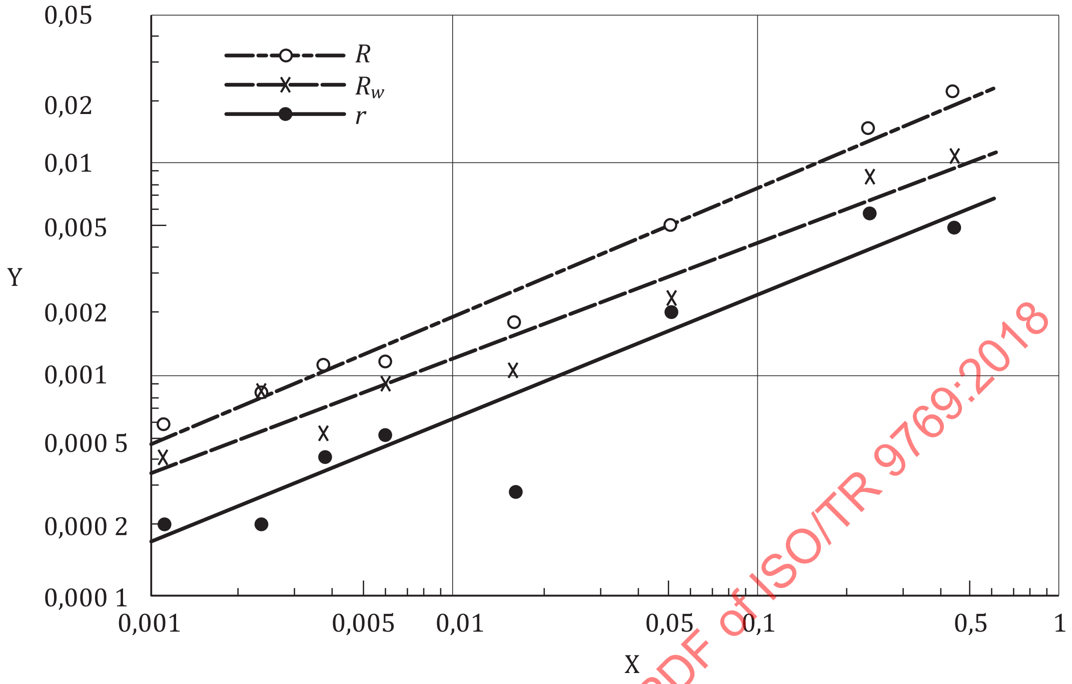
$\lg R_W = 0{,}849\,3 \lg \bar{w}_N - 1{,}137\,3$ Correlation coefficient = 0,982 3

$\lg R = 0{,}573\,9 \lg \bar{w}_N - 1{,}459\,7$ Correlation coefficient = 0,971 9

where $\bar{w}_N$ is the average nitrogen content, expressed as a percentage by mass, obtained from three determinations in each laboratory.

Figure A.34 — Logarithmic relationship between nitrogen content ($\bar{w}_N$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.14.2 Document: ISO 10702:1993, see Figure A.35.



Key

X nitrogen content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}579\,3 \lg \overline{w}_{N,1} - 2{,}045\,9$$

$$\lg R_W = 0{,}620\,4 \lg \overline{w}_{N,2} - 1{,}487\,4$$

$$\lg R = 0{,}539\,7 \overline{w}_{N,1} - 1{,}842\,2$$

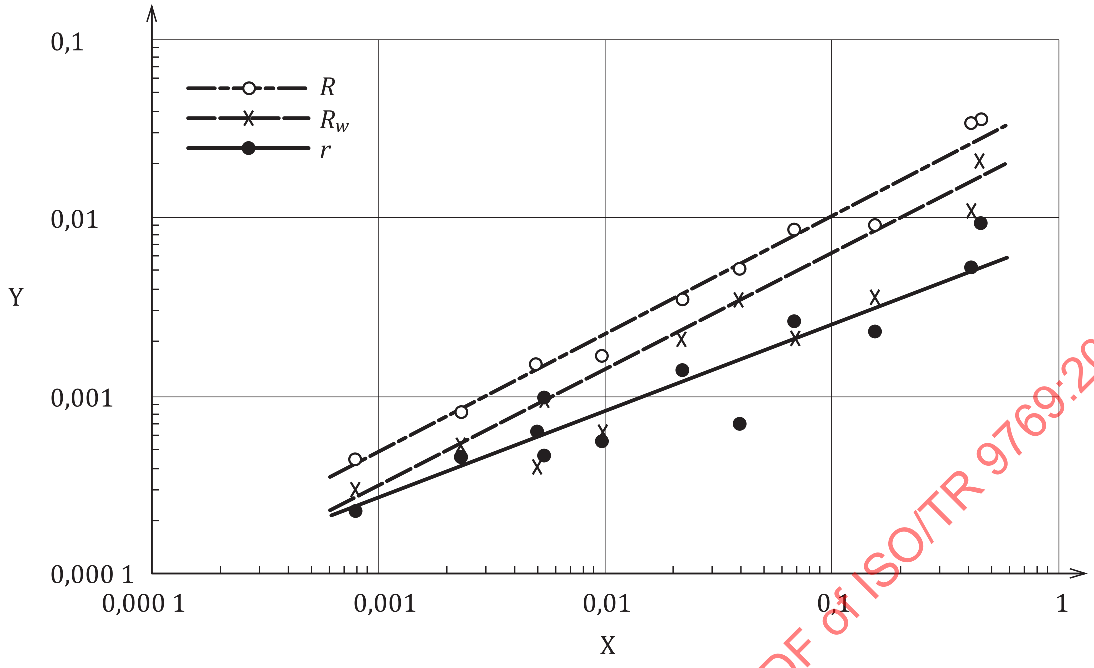
where

$\overline{w}_{N,1}$ is the average nitrogen content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{N,2}$ is the average nitrogen content, expressed as a percentage by mass, obtained between days.

Figure A.35 — Logarithmic relationship between nitrogen content ($\overline{w}_N$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.14.3 Document: ISO 10720:1997, see Figure A.36.



Key

X nitrogen content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

nitrogen contents up to 0,1 % (mass fraction)

$$\lg r = 0{,}433\,4 \lg \overline{w}_{\mathrm{N},1} - 2{,}271\,4$$

$$\lg R_W = 0{,}532\,6 \lg \overline{w}_{\mathrm{N},2} - 1{,}922\,9$$

$$\lg R = 0{,}665\,3 \lg \overline{w}_{\mathrm{N},1} - 1{,}354\,0$$

nitrogen contents between 0,1 % (mass fraction) and 0,5 % (mass fraction)

$$\lg r = 0{,}642\,4 \lg \overline{w}_{\mathrm{N},1} - 2{,}063\,1$$

$$\lg R_W = 0{,}838\,8 \lg \overline{w}_{\mathrm{N},2} - 1{,}617\,1$$

$$\lg R = 0{,}594\,8 \lg \overline{w}_{\mathrm{N},1} - 1{,}424\,7$$

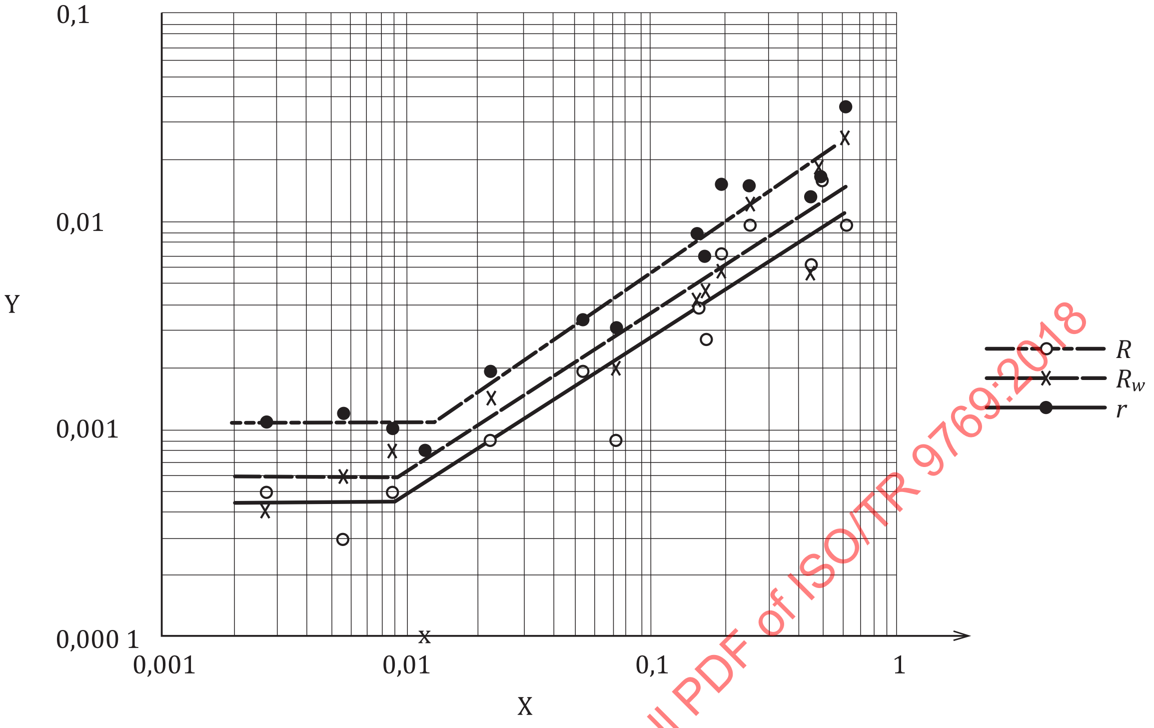
where

$\overline{w}_{\mathrm{N},1}$ is the average nitrogen content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{N},2}$ is the average nitrogen content, expressed as a percentage by mass, obtained between days.

Figure A.36 — Logarithmic relationship between nitrogen content ($\overline{w}_{\mathrm{N}}$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.14.4 Document: ISO 15351:1999, see Figure A.37.



Key

X nitrogen content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}7655 \lg \bar{w}_N - 1{,}782\ 2$$

$$\lg R_W = 0{,}763\ 9 \lg \bar{w}_N - 1{,}653\ 7$$

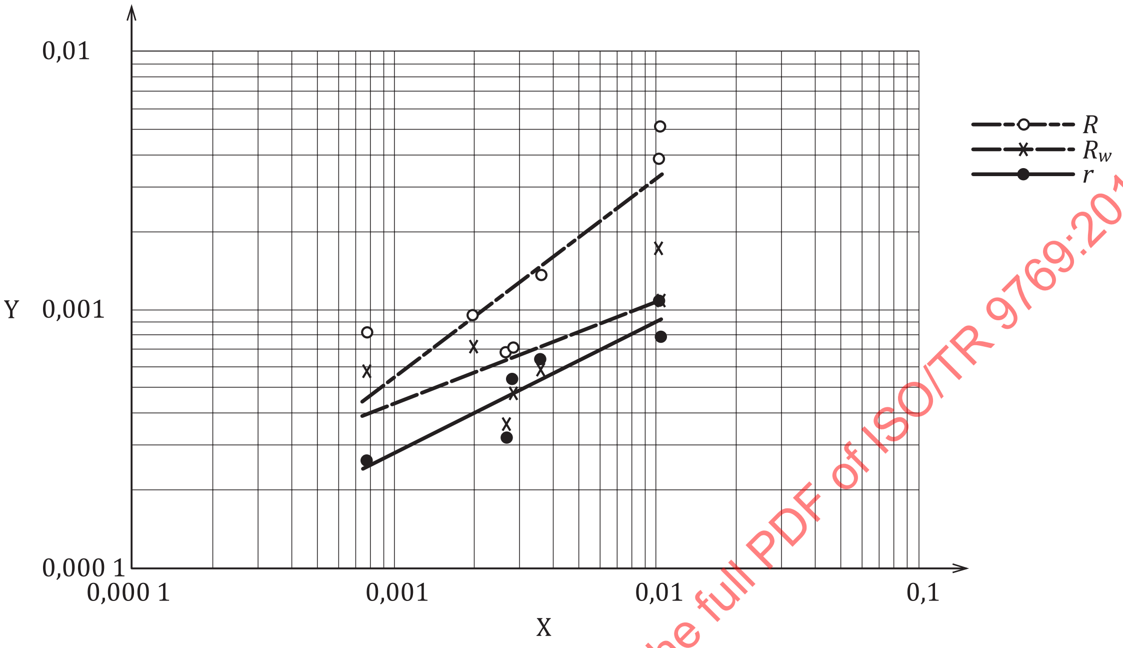
$$\lg R = 0{,}845\ 2 \lg \bar{w}_N - 1{,}405\ 6$$

where $\bar{w}_N$ is the average nitrogen content, expressed as a percentage by mass, obtained from three determinations in each laboratory.

Figure A.37 — Logarithmic relationship between nitrogen content ($\bar{w}_N$) and repeatability (r) and reproducibility (R and R_W)

A.15 Oxygen, O

Document: ISO 17053:2005, see Figure A.38.



Key

X oxygen content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

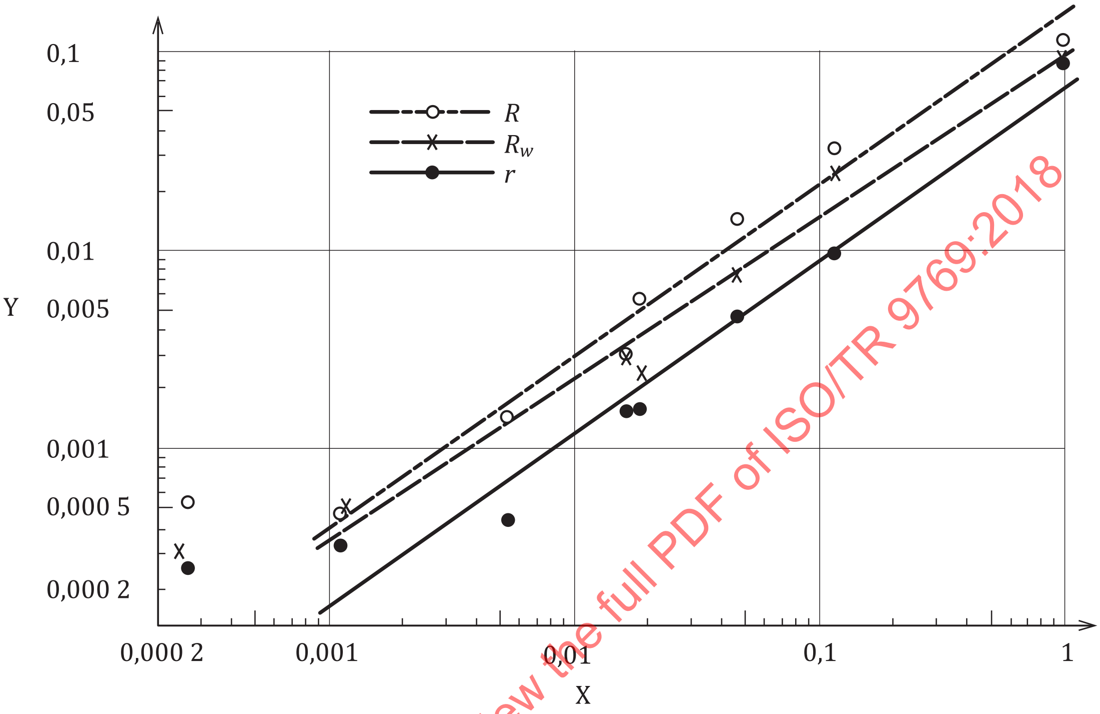
R_W within-laboratory reproducibility

r repeatability

Figure A.38 — Logarithmic relationship between oxygen content ($\bar{w}_O$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.16 Phosphorus, P

Document: ISO 10714:1992, see Figure A.39.



Key

X	phosphorus content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

$$\lg r = 0{,}874\,0 \lg \overline{w}_{\mathrm{P},1} - 1{,}166$$

$$\lg R_W = 0{,}809\,9 \lg \overline{w}_{\mathrm{P},2} - 1{,}023$$

$$\lg R = 0{,}860\,3 \lg \overline{w}_{\mathrm{P},1} - 0{,}812\,4$$

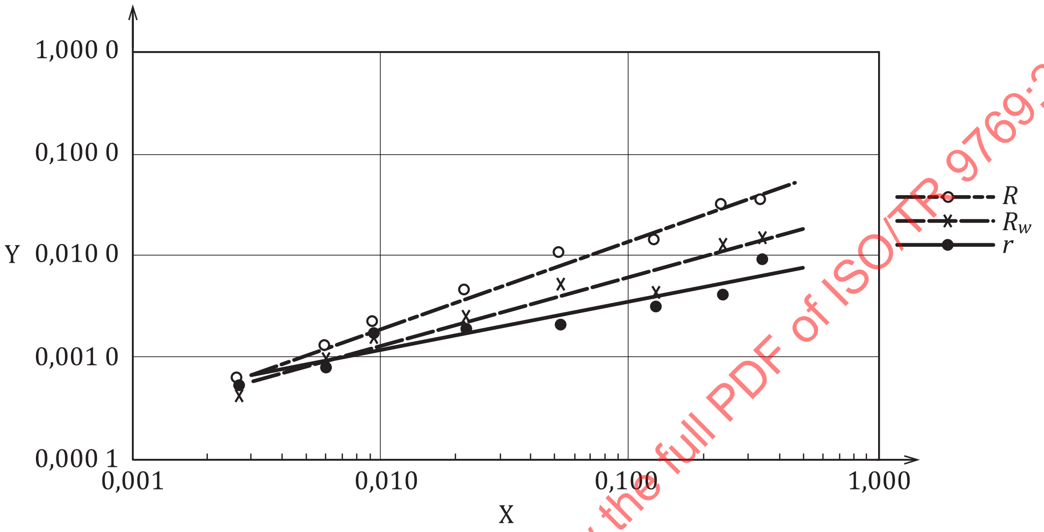
Figure A.39 — Logarithmic relationship between phosphorus content ($\overline{w}_{\mathrm{P}}$) and repeatability ($r$) and reproducibility ($R$ and R_W)

A.17 Sulfur, S

A.17.1 Document: ISO 671:1982.

There is no graphical representation of precision data in the standard.

A.17.2 Document: ISO 4934:2003, see Figure A.40.



Key

X sulfur content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}481\,2 \lg \overline{w}_{S1} - 1{,}972$$

$$\lg R_W = 0{,}675\,5 \lg \overline{w}_{S2} - 1{,}521\,3$$

$$\lg R = 0{,}862\,6 \lg \overline{w}_{S1} - 0{,}989$$

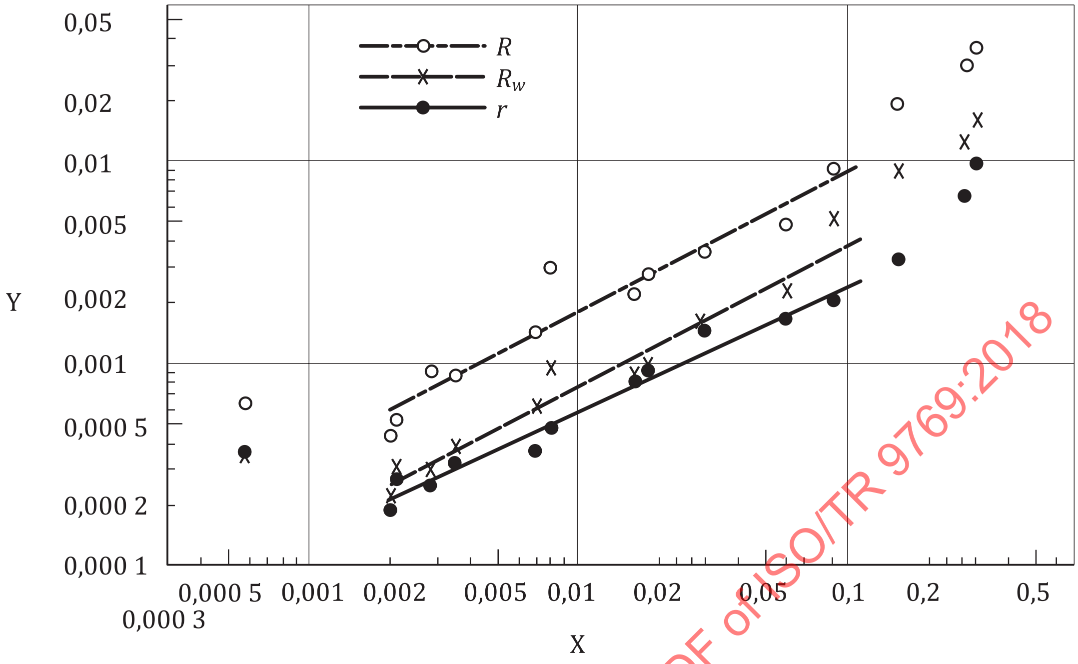
where

$\overline{w}_{S1}$ is the general mean within a day;

$\overline{w}_{S2}$ is the general mean between days.

Figure A.40 — Logarithmic relationship between sulfur content ($\overline{w}_S$) and repeatability (r) and reproducibility (R and R_W)

A.17.3 Document: ISO 4935:1989, see Figure A.41.



Key

X sulfur content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

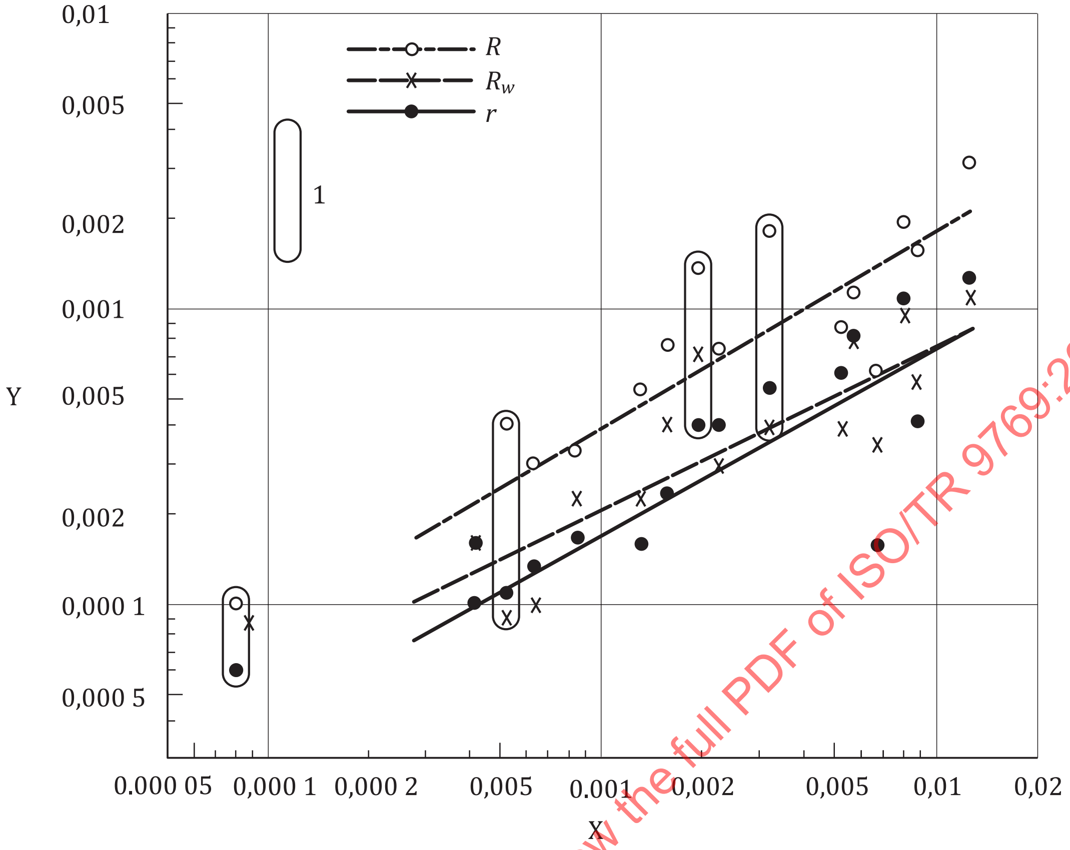
$$\lg r = 0{,}622\,12 \lg \overline{w}_S - 1{,}996\,5$$

$$\lg R_W = 0{,}700\,66 \lg \overline{w}_S - 1{,}709\,4$$

$$\lg R = 0{,}688\,78 \lg \overline{w}_S - 1{,}369\,3$$

Figure A.41 — Logarithmic relationship between sulfur content ($\overline{w}_S$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.17.4 Document: ISO 10701:1994, see Figure A.42.



Key

X sulfur content, % (mass fraction)
Y precision, % (mass fraction)
R reproducibility
R_W within-laboratory reproducibility
r repeatability
1 rejected

$$\lg r = 0{,}622\,4 \lg \bar{w}_{S1} - 1{,}895\,5$$

$$\lg R_W = 0{,}546\,7 \lg \bar{w}_{S2} - 2{,}036\,8$$

$$\lg R = 0{,}675\,6 \lg \bar{w}_{S1} - 1{,}400\,5$$

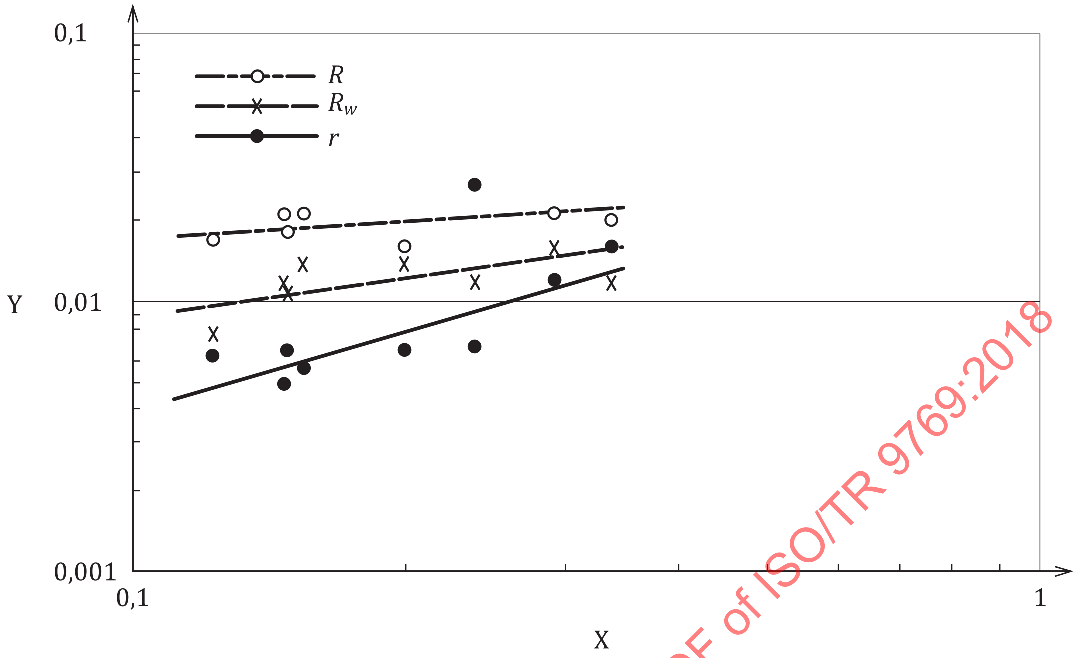
where

$\bar{w}_{S1}$ is the average sulfur content, expressed as a percentage by mass, obtained within a day;

$\bar{w}_{S2}$ is the average sulfur content, expressed as a percentage by mass, obtained between days.

Figure A.42 — Logarithmic relationship between sulfur content ($\bar{w}_S$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.17.5 Document: ISO 13902:1997, see Figure A.43.



Key

X	sulfur content, % (mass fraction)
Y	precision, % (mass fraction)
R	reproducibility
R_W	within-laboratory reproducibility
r	repeatability

$$\lg r = 0{,}951\,4 \lg \overline{w}_{S1} - 1{,}441$$

$$\lg R_W = 0{,}392\,8 \lg \overline{w}_{S2} - 1{,}624$$

$$\lg R = 0{,}177\,7 \lg \overline{w}_{S1} - 1{,}574$$

where

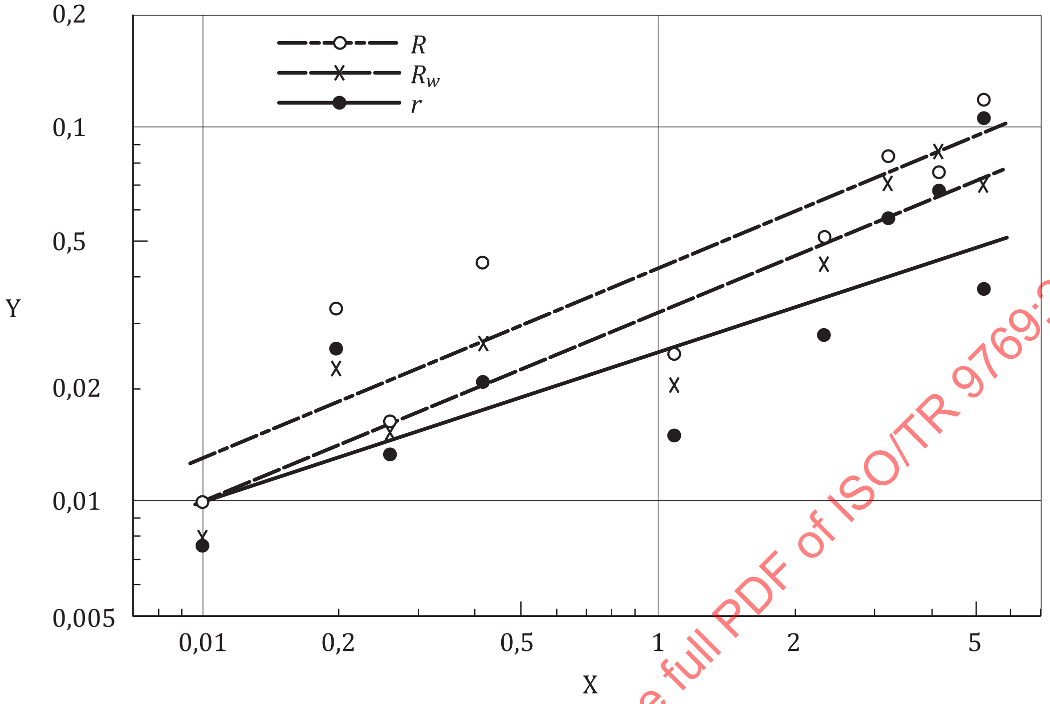
$\overline{w}_{S1}$ is the average sulfur content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{S2}$ is the average sulfur content, expressed as a percentage by mass, obtained between days.

Figure A.43 — Logarithmic relationship between sulfur content ($\overline{w}_S$) and repeatability (*r*) and reproducibility (*R* and R_W)

A.18 Silicon, Si

A.18.1 Document: ISO 439:1994, see Figure A.44.



Key

X total silicon content, % (mass fraction)

Y precision, % (mass fraction)

R reproducibility

R_W within-laboratory reproducibility

r repeatability

$$\lg r = 0{,}391\,8 \lg \overline{w}_{\mathrm{Si},1} - 1{,}586$$

$$\lg R_W = 0{,}498\,1 \lg \overline{w}_{\mathrm{Si},2} - 1{,}482$$

$$\lg R = 0{,}480\,5 \lg \overline{w}_{\mathrm{Si},1} - 1{,}377$$

where

$\overline{w}_{\mathrm{Si},1}$ is the average silicon content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{Si},2}$ is the average silicon content, expressed as a percentage by mass, obtained between days.

Figure A.44 — Logarithmic relationship between total silicon content ($\overline{w}_{\mathrm{Si}}$) and repeatability (*r*) and reproducibility (*R* and R_W)